Publication of BCSIR Laboratories, Dhaka

Publication of BCSIR Laboratories, Chittagong

Publication of BCSIR Laboratories, Rajshahi

Publication of Institute of Fuel research and Development (IFRD)

Publication of Institute of Food Science and Technology (IFST)

List of Publications of Dr. Md. Zahurul Haque, Director, IFST, BCSIR, Dhaka

Paper:

1. Animal feed from Bagasse. Part 1. - Chemical analysis of Bangladeshi Bagasse, **M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res**. XXI, No.(1-4),** 1986. p.80-88 (Accepted dt. 23.12.86).
2. Chemical investigations on Bangladeshi Turmeric. Part-1, Preparation of synthetic dyes from curcumin. **M.Z. Haque** and other. Bangladesh J. Sci. Ind. Res**. XXI, No.(1-4**), 1990. p.110-117 (Accepted dt. 16.9.89).
3. Chemical investigations on Bangladeshi Turmeric. Part-II, Substantivity of curcumin and its derivative dyes on Jute and Wool fibres. **M.Z. Haque** and other. Bangladesh J. Sci. Ind. Res. **XXV, No.(1-4),** 1990. p.143-152 (Accepted dt. 16.9.89).
4. Chemical analysis of Bangladeshi stick lac, seed lac, shellac, dewaxed shellac and bleached lac. **M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res. **XXV, Nos.(1-4),** 1990, p.206-208 (Accepted dt. 28.8.90).
5. Investigation on lac : Bleaching of lac. **M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res. **XXVII, Nos.(3-4),** 1992, p.134-140 (Accepted dt. 7.10.91).
6. Investigation on Lac : Preparation of varnish for copper wire insulation. **M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res. **XXIX, No.1**, 1994, p.64-69 (Accepted dt. 22.3.94).
7. Production of animal feed using sugarcane bagasse. **M.Z. Haque** and others. Rajshahi University Studies, Part-B, **Vol. 22,** 1994, p.1-9 (Accepted dt. 28.7.94).
8. Investigation on lac : Physico-chemical studies of lac waste (kiri). **M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res. **XXX, No.1**, 1995, 137-146 (Accepted dt. 7.3.94).
9. Investigation on lac : Isolation, utilization and analysis of lac-wax**. M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res. **XXX, No.(2-3),** 1995, p.47-54(Accepted dt. 14.9.94).
10. Investigation on lac : Effect of aging on solubility behaviour of seed lac, shellac, bleached lac and dewaxed shellac**. M.Z. Haque** and others. Bangladesh J. Sci. Ind. Res. **XXX, No.(2-3),** 1995, p.98-103 (Accepted dt. 4.5.94).
11. Reactions of diazonium salts with aryl dithiocarbamate. Part-I : Formation of phenyl azo phenyl dithiocarbamate. **M. Zahurul Haque** and others, Journal of Bangladesh Chemical Society, **8 (1),** 91-94, 1995 (Accepted dt. 20.9.94).
12. Synthesis of some NNSS metal complexes of 1, 4-dithia-6, 9-diza-5,10-di-iminocyclodecane. **M. Zahurul Haque** and others, Journal of Bangladesh Chemical Society, **9 (2),** 215-220, 1996 (Accepted dt. 4.11.96).
13. Synthesis of some NNSS metal complexes of bis-S-substituted phynyl-iosothiocarbamido ethane. **M. Zahurul Haque** and others, Journal of Bangladesh Academy of Sciences, Vol. **21,** No.2, 165-169, 1997 (Accepted dt. 23.7.97).
14. Isolation and purification of aleuritic acid and its esters from Bangladeshi shellac (Lac). **M. Zahurul Haque** & others. Journal of Bangladesh Chemical Society, **10 (1),** 93-97, 1997 (Accepted dt. 27.6.97).

15. Synthesis and characterization of some thiocarbamide derivatives, Part I : Synthesis of 1, 4-dithia-6, 9-diza-5, 10-di-iminocyclodecane. **M. Zahurul Haque** and others, Bangladesh J. Sci. Ind. Res., **32 (3),** 1997, p.464-466 (Accepted dt. 26.2.97).

16. Synthesis and characterization of some SSNN containing macrocyclic organic compounds. Pat I : Synthesis of bis-alkyl-di-dithiocarbamic acid derivatives. **M. Zahurul Haque** and others, Journal of Bangladesh Chem. Soc.,**10(2),**159-164,1997 (Accepted dt. 15.9.97).

17. Synthesis and characterization of some SSNN containing macrocyclic organic compounds. Pat II : Synthesis of bis-aryl-di-dithiocarbamic acid derivatives**. M. Zahurul Haque** and others, Journal of Bangladesh Chem. Soc., **10(2),** 165-169, 1997 (Accepted dt. 30.7.97).

18. Investigation on lac : Effect of curing agent on life of lac (Shellac) under heat, **M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **11 (1 & 2),** 123-127, 1998 (Accepted. dt 16.8.98).

19. Investigation on Bangladeshi lac dye. Part I - Isolation and purification of laccaic acid A_1 from stick lac**. M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **11 (1 & 2),** 129-134, 1998 (Accepted dt. 15.10.98).

20. Investigation on lac : Physico-chemical studies of polymerized seed lac, shellac and dewaxed shellac. **M.Z. Haque** and others, Bangladesh J. Sci. Ind. Res., 3**3(1), 55-58,** 1998 (Accepted dt. 22.1.96).

21. Investigation on lac : Solubility behaviour of thermally polymerized seed lac, shellac and dewaxed shellac. **M.Z. Haque** and others, Bangladesh J. Sci. Ind. Res. **33 (1), 103-106,**1998 (Accepted dt. 3.6.96).

22. Chemical modification of lac. Part I - Some ethylene-glycol modifications and its derived products. **M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **12 (1),** 93-98, 1999 (Accepted dt. 18.4.99).

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25. Chemical investigation on thermally polymerized aleuritic acid**. M. Zahurul Haque** and others. Journal of Bangladesh Academy of Sciences, **Vol. 24, No. 2**, 59-65, 2000 (Accepted dt. 30.1.2000).

26. Synthesis and characterization of some thocarbamide derivatives. Part II: Synthesis of bis-s-substituted phenylisothiocarbamidoethane. **M. Zahurul Haque** and others, Bangladesh J. Sci. Ind. Res., **36 (1-4),** 148-150, 2001(Accepted dt. 11.7.2000).

27. Reactions of diazonium salts with phenyl-dithiocarbamate. Part II: Formation of related aryl azo phenyldithiocarbamates. **M. Zahurul Haque** and others. Journal of Indian Chemical Society, **Vol. 78**, No.7, 2001, 372-373 (Accepted dt. 29.12.2000).

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29. Synthesis of some new thiocarbamides from a constituent of lac and studies on their antimicrobial activities. **M. Zahurul Haque** and others. Journal of Indian Chemical Society, **Vol. 79,** No. 10, 2002, 841-842. (Accepted dt. 24.4.2002).

30. Investigation on lac: Preparation of ready-made varnish. **M. Z. Haque** and others. Bangladesh J. Sci. Ind. Res., **36 (1-4),** 163-166, 2001(Accepted dt. 12.9.2002).

31. Production of dewaxed shellac flakes and its specification. **M. Z. Haque** and others. Bangladesh J. Sci. Ind. Res., **37,** 131-136, 2002 (Accepted dt. 18.10.2003).

32. Synthesis and biological activity of some new thiocarbamides. **M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **16(2),** 175-178, 2003 (Accepted dt. 17.12.2003).

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36. Synthesis of nickel (II) complexes with some thiocarbamide derivatives. **M. Zahurul Haque** and others. Journal of Indian Chemical Society, **82,** 401-403, 2005, (Accepted dt. 8.2.2005).

37. Mass fragmentation pattern of some mono- and di- substituted formadines: Part 1- Mass pattern of 1-arylformadines. **M. Zahurul Haque** and others. Pakistan Journal of. Scientific and Industrial Research, **48 (6),** 386-388, 2005 (Accepted dt. 31.3.2005).

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41. Feasibility studies on the production of lac crop in Bangladesh and its diversified application in industrial sector. **M. Zahurul Haque**. Proceedings Bangladesh Chemical Congress-2004, 329-332. Bangladesh Chemical Society. ISBN 984-32-3488-X.

42. Investigations on *Terminalia arjuna* fruits: Part 1- Isolation of compounds from petroleum ether fractions. **M. Zahurul Haque** and others. Bangladesh J. Sci. Ind. Res. **43(1),** 123- 130, 2008 (Accepted dt. 21.1.07).

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44. Extraction of henna leaf dye and its dyeing effects on textile fibre. **M. Zahurul Haque** and others. Bangladesh J. Sci. Ind. Res. **42(2),** 217-222, 2007 (Accepted dt. 8.4.07).

45. Studies on the stabilization of rice bran and its oil (part II). **M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **19(1 & 2),** 45-50, 2006 (Accepted dt. 7.2.2007).

46. Studies on cyanex-272 complexes of Mg (II), Ca (II) and Fe (III). **M. Zahurul Haque** and others. Bangladesh J. Sci. Ind. Res. **42(4),** 475-482, 2007 (Accepted dt. 7.10.07).

47. Studies on the isolation and biological activities of chloroform extracts of *Terminalia arjuna* fruits. **M. Zahurul Haque** and others. Bangladesh J. Life Sci. **19(2),** 103-107, 2007 (December) (Accepted dt. 12.9.07).

48. Synthesis and biological activities of some new zolidine derivatives from 1-substituted-3-N-aleuritamido thiocarbamides. **M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **20(1),** 99- 103, 2007 (Accepted dt. 17.12.07).

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51. Study of cytotoxic activity of *Aloe vera* L. leaf extracts. **M. Zahurul Haque** and others. Bangladesh J. Life Sci. **20(2):**141-145, 2008 (December) (Accepted dt. 16.8.2008).

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53. Studies on the fatty acids, lipids and glyceride compositions of pitraj (*Amoora rohituka*) seed oil. **M. Zahurul Haque** and others. Journal of Bangladesh Chemical Society, **21(2),** 155-160, 2008 (Accepted dt. 21.7.2009).

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55. Extraction of alkaloids and oils from karanja (*Pongamia pinnata*) seed. **M. Z. Haque** and others. *J. Sci. Res*. **3 (3),** 669-675 (2011).

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57. Studies on the production of musabbar from *Aloe vera*. **M. Zahurul Haque** and others. *Journal of Advanced Scientific Research, 2012;3(1): 51 - 54* .

58. Physico-chemical characteristics, proximate composition, vitamins, minerals and protein contents of some selected non-conventional oil cakes in Bangladesh. **M. Z. Haque** and others. *Bangladesh J. Life Sci., 23(2):51-59, 2011 (December)*.

59. Production of kalomegh syrup and studies on its toxic activities. **M. Zahurul Haque** and others. *Journal of Advanced Scientific Research, 2012;3(2)*:45-48.

60. Insecticidal activity of plant extracts against Tribolium castaneum Herbst. **M. Zahurul Haque** and others. *Journal of Advanced Scientific Research, 2012;3(3):80-84*.

61. Phyto-chemical and anti-bacterial screening of musabbar prepared from *Aloe vera*. **M. Zahurul Haque** and others. *Journal of Advanced Scientific Research, 2012;3(4):74 – 77.*

62. Phyto-chemical screening and anti-bacterial activity studies on *Leea macrophylla* seeds extracts. **M. Z. Haque** and others. *Journal of Scientific Research,5(2), 399-405 (2013).*

63. Preparation and application of different size materials on the cotton yarn and investigating the effect of sizing on the tensile properties of cotton yarn. M. Zahurul Haque and others. *Bangladesh J. Sci. Ind. Res.*- In Press-2014.

64. Proximate analysis of Aloe vera leaves. **M. Zahurul Haque** and **others.** *IOSR Journal of Applied Chemistry (IOSR-JAC), e ISSN: 2278-5736. Volume 7, Issue 6 Ver. I. (Jun. 2014), pp26-40.*
65. Design and development of a process for the production of aloe lemon drink and studies on its shelf life. **M. Zahurul Haque** and others. *International Journal of Emerging Technology and Advanced Engineering, Volume 5, Issue 7, July 2015, pp 385-389.*
66. Evaluation of njutritional properties of some small indigenous fishes species in Bangladersh. **M. Zahurul Haque** and others. *International Journal of Biosciences, Vpl. 6,No. 6, pp 102-109, 20105.*
67. Comparative bioactove compounds determination and in-vitro anti-oxidant properties of newly developrd soy mixed wheat flour and traditional wheat flour. *International Journal of Food Properties-Accepted for publication dt. 15October 2015.*
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Process:

69. A process for the production of animal feed from sugarcane bagasse. Accepted for leasing out vide letter No. Sec/PPP/1(162)/82 (3)/1600 dt. 12.12.94.
70. A process for the production of dewaxed lac. Process developed and pilot plant studies completed-1996.
71. A process for the production of bleached lac from seed lac **(Leased out)**. Accepted for leasing out vide letter No. Sec/PPP/62-113/97/1054 dt. 22.09.97.
72. A process for the production of lac-based capping cement. Accepted for leasing out vide letter No. Sec/PPP/62-130/97/2833 dt. 01.09.97.
73. A process for the production of dewaxed lac powder from seed lac **(Leased out)**. Accepted for leasing out vide letter No. Sec/PPP/62-130/97/2834 dt. 01.09.97
74. A process for the production of adhesive based on urea - formaldehyde resin. Accepted for leasing out vide letter No. Sec/PPP/62-130/97/2834 dt. 01.09.97.
75. A process for the production of printing ink. Accepted for leasing out vide letter No. Sec/PPP/62-226/2002/638 dt.7.4.2003.
76. A process for the production of drawing ink from seed lac. Accepted for leasing out vide letter No. Sec/PPP/62-225/2002/640 dt.7.4.2003.
77. A process for the production of dewaxed shellac flakes from seed lac. Accepted for leasing out vide letter No. Sec/PPP/62-235/2003/649 dt.7.4.2003.
78. A process for the production of ready-made transparent wood varnish from lac. Accepted for leasing out vide letter No. Sec/PPP/62-234/2003/853 dt.20.4.2003.
79. A process for the production of dewaxed shellac-molasses modified resin. Accepted for leasing out vide letter No. Sec/PPP/62-256/2003/615 dt.11.8.2003.
80. A process for the production of lac-oil modified product. Accepted for leasing out vide letter No. Sec/PPP/62-255/2003/613 dt.11.8.2003.
81. A process for the production of hydrolysed lac-molasses modified resin. . Accepted for leasing out vide letter No. Sec/PPP/62-257/2003/612 dt.11.8.2003.

82. A process for the production of deep brown shade on silk fabric and yarn with khair (catechu brown dye). Accepted for leasing out vide letter No. Sec/PPP/62-269/2003/620 dt.11.8.2003.

83. A process for the production of reddish brown shade on silk fabric and yarn with blended vegetable dye (khair and dabshell dye). Accepted for leasing out vide letter No. Sec/PPP/62-260/2003/618 dt.11.8.2003.

84. A process for the production of orange red shade on silk fabric with blended vegetable and synthetic dye (dabshell dye and methyl orange dye). Accepted for leasing out vide letter No. Sec/PPP/62-262/2003/638 dt.12.8.2003.

85. A process for the production of deep red shade on silk fabric with blended vegetable and synthetic dye (dabshell dye and reactive red dye). Accepted for leasing out vide letter No. Sec/PPP/62-263/2003/621 dt.11.8.2003.

86. A process for the production of hardboard from bagasse. Accepted for leasing out vide letter No. Sec/PPP/62-258/2003/1285 dt.21.3.2004.

87. A process for the production of dry pineapple slices. Accepted for leasing out vide letter No. Sec/PPP/62-349/2004/1800 dt.26.4.2004.

88. A process for the production of chalta chutney. Accepted for leasing out vide letter No. Sec/PPP/62-348/2004/1801 dt.26.4.2004.

89. A process for the production of sweet mango pickle. Accepted for leasing out vide letter No. Sec/PPP/62-344/2004/1802 dt.26.4.2004.

90. A process for the production of green mango pulp. Accepted for leasing out vide letter No. Sec/PPP/62-346/2004/1803 dt.26.4.2004.

91. A process for the production of mango chutney. Accepted for leasing out vide letter No. Sec/PPP/62-345/2004/1804 dt.26.4.2004.

92. A process for the production of dry mango slices. Accepted for leasing out vide letter No. Sec/PPP/62-347/2004/1805 dt.26.4.2004.

93. A process for the production of gastrointestinal hyperacidity ulceration curative agent **(Leased out)**. Accepted for leasing out vide letter No. Sec/PPP/62-390/2004/3377 dt.4.8.2004.

94. A process for the production of sinus and nasal obstruct curative agent. Accepted for leasing out vide letter No. Sec/PPP/62-391/2004/3378 dt.4.8.2004.

95. A process for the production of liver curative agent. Accepted for leasing out vide letter No. Sec/PPP/62-389/2004/3380 dt.4.8.2004.

96. A process for the production of musabbar from *Aloe vera* **(Leased out)**. Accepted for leasing out vide letter No. Sec/PPP/62-388/2004/3566 dt.15.8.2004.

97. A process for the production of tomato powder. Accepted for leasing out vide letter No. Sec/PPP/62-404/2004/1350 dt. 21.11.2004.

98. A process for the production of mango squash. Accepted for leasing out vide letter No. Sec/PPP/62-403/2004/1351 dt. 21.11.2004.

99. A process for the production of vitamin enriched medicated amrha jelly. Accepted for leasing out vide letter No. Sec/R&D Div./62-433/2004/172 dt. 10.1.2005.

100. A process for the production of *Aloe vera* syrup **(Leased out)**. Accepted for leasing out vide letter No. Sec/R&D Div./62-423/2004/173 dt. 10.1.2005.

101. A process for the production of natural pesticide for stored grains from a herbal source. Accepted for leasing out vide letter No. Sec/R&D Div./62-435/2004/174 dt. 10.1.2005.

102. A process for the production of nutritious palm biscuit. Accepted for leasing out vide letter No. Sec/R&D Div./62-543/2006/473 dt. 20.8.2006.

103. A process for the production of herbal tulsi tea. Accepted for leasing out vide letter No. Sec/R&D Div./62-542/2006/472 dt. 20.8.2006.

104. A process for the production of palm powder. Accepted for leasing out vide letter No. Sec/R&D Div./62-564/2006/888 dt. 25.2.2007.

105. A process for the production of kalomegh pearl. Accepted for leasing out vide letter No. Sec/R&D Div./62-563/2006/993 dt. 1.8.2007.

106. A process for the production of cracked heel cream (**Leased out**). Accepted for leasing out vide letter No. Sec/R&D Div./62-574/2007/22 dt. 3.6.2007.

107. A process for the production of Aloe vera powder (**Leased out**). Accepted for leasing out vide letter No. Sec/R&D Div./62-573/2007/23 dt. 3.6.2007.

108. A process for the production of anti-fungal ointment from herbal source (**Leased out**). Accepted for leasing out vide letter No. Sec/R&D Div./62-572/2007/329 dt. 21.6.2007.

109. A process for the production of herbal medicated hair dye. Accepted for leasing out vide letter No. Sec/R&D Div./62-480/2005/2275 dt. 30.10.2007.

110. A process for the production of cabbage chutney. Accepted for leasing out vide letter No. Sec/R&D Div./62-460/2005/1277 dt. 13.3.2008.

111. A process for the production of vitamin enriched karamcha squash. Accepted for leasing out vide letter No. Sec/R&D Div./62-608/2007/1279 dt. 13.3.2008.

112. A process for the production of nutritious palm cake. Accepted for leasing out vide letter No. Sec/R&D Div./62-598/2007/1282 dt. 13.3.2008.

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115. A process for the production of high quality ball point pen black ink. Accepted for leasing out vide letter No. Sec/R&D Div./62-551/2006/1 dt. 1.6.2008.

116. A process for the production of insulating varnish. Accepted for leasing out vide letter No. Sec/R&D Div./62-505/2005/686 dt. 7.7.2008.

117. A process for the production of aloe lemon drink **(Leased out)**. Accepted for leasing out vide letter No. Sec/R&D Div./62-653/2008/848 dt. 16.7.2008.

118. A process for the production of herbal aloe shampoo **(Leased out)**. Accepted for leasing out vide letter No. Sec/R&D Div./62-610/2007/1345 dt. 20.8.2008.

119. A process for the production of aloe toothpaste. Accepted for leasing out vide letter No. Sec/R&D Div./62-650/2008/2317 dt. 26.10.2008.

120. A process for the production of *Pongamia pinnata* seed oil. Accepted for leasing out vide letter No. Sec/R&D Div./62-675/2008/68 dt. 6.1.2009.

121. A process for the production of gulancha starch. Accepted for leasing out vide letter No. Sec/R&D Div./62-651/2008/2046 dt. 24.5.2009.

122. A process for the production of condensed palm pulp. Accepted for leasing out vide letter No. Sec/R&D Div./62-705/2008/355 dt. 21.7.2009.

123. A process for the production of high longevity candle. Accepted for leasing out vide letter No. Sec/R&D Div./62-725/2009/356 dt. 21.7.2009.

124. A process for the production of instant amloki powder drink. Accepted for leasing out vide letter No. Sec/R&D Div./62-711/2008/357 dt. 21.7.2009.

125. A process for the production of aloe vera tablet. Accepted for leasing out vide letter No. Sec/R&D Div./62-739/2009/1557 dt. 13.10.2009.

126. A process for the production of herbal cough syrup **(Leased out)**. Accepted for leasing out vide letter No. Sec/R&D Div./62-706/2008/1556 dt. 13.10.2009.

127. A process for the production of karamcha sauce. Accepted for leasing out vide letter No. Sec/R&D Div./62-707/2008/1555 dt. 13.10.2009.

128. A process for the production of aloe vera vanishing cream (**Leased out**). Accepted for leasing out vide letter No. Sec/R&D Div./62-743/2009/443 dt. 24.01.2010.

129. A process for the production of shatamuli powder derink (**Leased out**). Accepted for leasing out vide letter No. Sec/R&D Div./62-767/2008/1161 dt. 1.03.2010.

130. A process for the production of high quality printing ink (red). Accepted for leasing out vide letter No. 39.373.037.01.00.003.2010/4445 dt. 19.08.2010.

131. A process for the production of super paper adhesive. Accepted for leasing out vide letter No. 39.373.037.01.00.004.2010.5505 dt. 25.10.2010.

132. A process for the production of herbal burning soothing agent. Accepted for leasing out vide letter No. Sec/R&D Div./62-652/2008/8038 dt. 12.12.2010.

133. A process for the production of super wood adhesive (**Leased out**). Accepted for leasing out vide letter No. 39.373.037.01.00.00.007.2010.721 dt. 7.2.2011.

134. A process for the production of printing roller wash. Accepted for leasing out vide letter No. 39.373.037.01.00.048.2010.724 dt. 7.2.2011.

135. A process for the production of photographic film developer. Accepted for leasing out vide letter No. 39.373.037.01.00.001.2010.2568 dt. 2.6.2011.

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LIST OF PUBLICATION OF MD. NAZIM UDDIN, SO, IFST, BCSIR.

I have **thirteen** international and **one** national scientific paper.

1. Md. Nazim U; Rahman MA et al. Chromatographic (HPLC) analysis explores the potential antioxidative agents of Argyreia argentea var. discolor (Blume) Forman extract'. Journal of Pharmacy and Bioallied Sciences 2016 (ACCEPTED)

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Quality Study of Rice Husk Ash (RHA) Brick Using Neutron Radiography Technique

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Abstract

A powerful non-destructive testing (NDT) technique is adopted to study the quality of RHA brick-1 and RHA brick-2. In that case, rice husk ash has been utilized for the preparation of bricks in full replacement of clay. In these studies, homogeneity of elemental distribution, water absorption and size and shape of the pores have been observed. From the studies, it was observed that elemental distribution is very good at various level, large number of porosity is presented with little bit size, initial rate of absorption (IRA) due to first five minutes immersion of water is higher compared to other immersion time (>5 minute), incremental water intrusion area increases very slowly with the increasing immersion time and the water absorption for RHA brick-2 becomes saturated very early than that of the RHA brick-1.

Keywords

Neutron Radiography, RHA, IRA and Water Absorption Behavior

1. Introduction

Rice husks are the hard protecting coverings of grains to protect rice at the ripening period. A large number of ashes is produced at the burning time which is used as a waste and pollute environment and it is also a great environment threat causing damage to land and stored/dumped area. On average 20% of the rice paddy is husk, giving an annual total production of 120 million tones [1]. Because of its low density, it is very difficult to transfer from one place to another place for cleaning residential area. Carbon which is produced due to burning

of the hush, is one of the greatest threats to the environment. From this point of view, it is necessary to proper manage the rise husk as a socio economic use. The ash itself (87 - 97)% is silica [2], highly porous and light weight, with a very high external surface area. Various factors which influence ash properties are incinerating conditions (temperature and duration), rate of heating, burning technique, crop variety and fertilizer used [3]. Apart from its use as a fuel in power plant, formation of produced carbon [4], a source of silica and silicon compounds [5] [6] and also for internal decorative bricks (which is quite newly vision).

The neutron radiography method/technique is a simple process of exposing some objects to an X-ray, gamma-ray, neutron beam and some other types of radiation and then attenuated out going beam from the object is passing through a special type of photographic film to form images of the objects on the radiographic film or detector. This method is also used for different research purposes such as study of water absorption and internal defects in jute reinforced biopol composite [7]; internal structure, internal defects in automated machine made environmentally friendly brick and conventionally made brick samples [8]-[10], different ceramic samples [11], different tiles [12], different building materials [13]; internal defects in electronic components [14]; water uptake and internal defects of jute reinforced polymer composite [15]; water absorption behavior in biopol and jute reinforced biopol composite [16] etc. It is a technique of making a picture of the internal details of the test object. Because of high penetrating power of neutron beam, neutron radiography technique is the best to measure any homogeneity, internal voids, cracks in the sample than the X-ray and gamma ray radiography. In this paper we have analysis the internal homogeneity, voids, cracks and water uptake behavior and uses of rice husk ash (RHA) brick, and also attempt has been made to collect data and information from various research work related to RHA using neutron radiography method.

2. Experimental Details

2.1. Sample Preparation

Rice husk ash is obtained by burning rice husk. Firstly rice husk ash is collected from the local rice production industry. After that rice husk ash and gum/one type of resin mixed together with optimum proportion of water. This mixer is inserted into water for a day to make solution. The next day to make sand-gum-husk ash-water solution a certain proportion of sand is mixed with this solution and stir homogeneously. Now, this solution stores into the same container for another one day. Now transfer and store this solution into respective mold and press by the weight of 10 ton. Now put out from the mold and dry another 28 days into normal ambient temperature or in an open air and obtained special type of rice husk ash (RHA) bricks. The elemental ratio of gum: water: husk ash: sand into the RHA brick is 1:3:9:110.

2.2. Loading Converter Foil and Film in to the NR-Cassette

A thin converter (gadolinium metal foil of 25 μm thickness) was placed at close contact with the emulsion surface of the X-ray industrial film. The loading of the X-ray industrial film (Agfa structurix D_4DW) into the NR cassette (18 cm × 24 cm) is a simple procedure [17] which requires a darkroom.

2.3. Placing of Sample and the NR-Cassette in the Experimental Facility

The RHA brick is placed in close contact with the NR cassette (shown in Figure 1) on the sample holder table. The NR cassette is placed on the cassette holder table.

2.4. Determination of Exposure Time

Exposure means passing of neutron beam through a sample and holding it onto a special film (Structurix, D4DW industrial film) in order to create a latent internal image of an object in the emulsion layers of that film. Exposure time differs for different samples, depending on the intensity of the neutron beam, neutron cross-section, density and thickness of the sample. The optimum exposure time of the dry RHA brick was determined by taking a series of neutron radiographs with different exposure time, while the reactor was operated at constant power 2.4 MW. In the present experiment the optimum exposure time is found 08 minutes for each RHA bricks. The samples were then irradiated for that optimum exposure time to obtain good neutron radiographic images of each RHA bricks one by one.

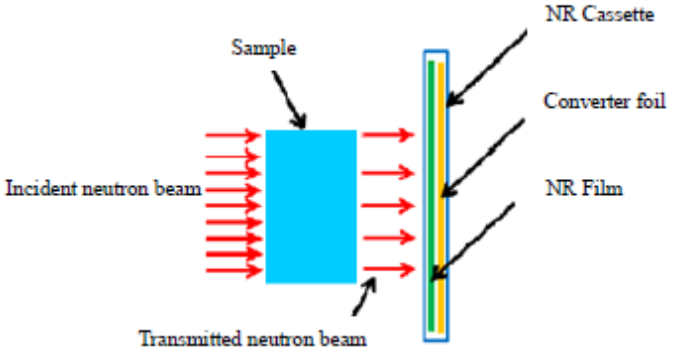

Figure 1. Diagram of showing neutron beam direction, placing of sample, NR film and converter foil inside the NR cassette.

2.5. Immersion Procedure of the RHA Brick Sample

The RHA brick is placed in a plastic pan and a constant 1.0 cm height of water level is maintained. The water level is observed very carefully and adds extra water to maintain water level during the immersion time. After 1, 3, 5 and 8 minutes immersion time the RHA brick sample taken off from the pan and extra water of outer surface of the RHA sample is removed by the tissue paper properly.

2.6. Obtained Radiographic Images

To obtain the neutron radiographic images of the dry and wet bricks the following procedures are completed.

1) Irradiation: After putting the RHA brick on the sample holder table the neutron beam was disclosed for 8 minutes by removing the wooden plug, lead plug and beam stopper from the front side of the beam collimator.

2) Developing: Developing is an image processing technique by which the latent image is converted into a metallic silver image [18]. Developing process is completed at 20˚C - 22˚C solution temperature for 7 minutes.

3) Fixing: The fixation solution will dissolve the unexposed silver-halide crystals leaving only the silver grains in the gelatin of the exposed film. The fixing is completed within a 5 minutes and control the fixture temperature at 20˚C - 22˚C.

4) Washing: Between the process of developing and fixing this radiographic film is washed for 1 minute at flowing tap water.

5) Final washing: The silver compound was formed during the fixing stage must be removed, since they can affect the silver image at the latter stage. For this reason the exposed film must be washed thoroughly in flowing tap water for 15 minutes after completion of fixing process of the exposed film. In that case water temperature is maintained at 20˚C - 22˚C also.

6) Drying: After final washing, the imaging films were dried by clipping in a hanger at fresh air/or in a drying cabinet to obtain final radiographic images of the dry and wet RHA bricks.

3. Mathematical Formulation

The neutron radiographic image represents the attenuating behavior of thermal neutron beam due to dry and wet RHA bricks. Attenuation of thermal neutron beam is mainly due to scattering and absorption interactions of neutrons with atomic nuclei. This attenuating response for dry RHA bricks can be written [19] as:

$$I = I_0 e^{-\mu_s t_s} \tag{1}$$

Here I and I_0 are the attenuated and incident neutron intensities (n/cm^2/sec) respectively, μ_s is the neutron attenuation coefficient (cm^{-1}) of the dry RHA bricks and t_s is the corresponding thickness (cm).

In case of wet RHA bricks the above equation can be written as follows:

$$I' = I_0 e^{-(\mu_s t_s + \mu_w t_w)} \tag{2}$$

where I' is the attenuated neutron intensity of wet RHA brick, μ_w is the neutron attenuation coefficient of

water and t_w is the thickness of the water absorbed by the RHA bricks.

From Equation (1) and Equation (2) the thickness of the absorbed water by the bricks can be calculated as follows:

$$t_w = -\ln(I'/I)/\mu_w \tag{3}$$

The gray value/neutron intensity of the radiographic images of the sample is changed with the increase of water absorbed by the samples. The attenuated neutrons beam enters the detector that resists the fraction of initial radiation intensity that has been transmitted by each point of the object and is then recorded by the radiographic film *i.e.* image detector.

The neutron intensity before reaching the RHA brick sample (object) is different from the intensity of the neutron after passing through the samples. The relationship between these two intensities is expressed through Equation (1). On the other side of the film, a light sensor (photocell) converts the penetrated light into an electrical signal. Actually, optical density is the darkness or opaqueness of a transparency film and is produced by film exposure and chemical processing. An image contains areas with different densities that are viewed as various shades of gray.

4. Gray Value

The visual appearance of an image is generally characterized by two properties such as brightness and contrast. Brightness refers to the overall intensity level and is therefore influenced by the individual gray-level (intensity) values of all the pixels within an image. Since a bright image (or sub image) has more pixel gray-level values closer to the higher end of the intensity scale, it is likely to have a higher average intensity value. Contrast in an image is indicated by the ability of the observer to distinguish separate neighboring parts within an image. This ability to see small details around an individual pixel and larger variations within a neighborhood is provided by the spatial intensity variations of adjacent pixels, between two neighboring sub images or within the entire image. Thus, an image may be bright (due to, for example, overexposure or too much illumination) with poor contrast if the individual target objects in the image have optical characteristics similar to the background. At the other end of the scale, a dark image may have high contrast if the background is significantly different from the individual objects within the image, or if separate areas within the image have very different reflectance properties.

An image that contains pixels with brightness values spread over the entire intensity scale is likely to have better contrast than the image with pixel gray-level values located within a narrow range. The relationship between the intensity spread at the pixel level and the overall appearance of an image provides the basis for image enhancement by gray-level transformation. The terms gray value and intensity are used synonymously to describe pixel brightness.

5. Image Detector

Silver chloride, bromide and iodide are collectively known as silver halides. These are used in various proportions in the radiographic film emulsions. In the manufacture of the radiographic emulsion the silver halide is formed as a dispersion of extremely small particles (microcrystal) in gelatin. When coated on a glass or film base and allowed to dry, this becomes a thin, tough, transparent layer which is very sensitive to short wavelengths of light. During the neutron exposure time electron is allowed to fall on the emulsion surface and its energy is absorbed by silver halide particles, causing local disruptions of some bonds that hold the crystalline structure together and release free silver atoms within the body of the crystal. Above a certain critical energy enough silver atoms are released to form a metallic silver or latent image. The developer is used to turn the latent image into a visible photographic image in metallic silver. This solution containing a reducing agent is capable of reducing silver halide to silver, but only in the case of those crystals which bear a latent image. The silver halide emulsion radiographic film is used as an image detector.

6. Results and Discussion

6.1. Porosity/Voids and Homogeneity Measurement

The quality of a HRA brick samples depends on the proper distribution of the contents, porosity, water absorp-

tion behavior etc. in the sample. The porosity, elemental distribution of the samples has been studied by measuring gray value/intensity ratio from the neutron radiographic images of each sample. Figure 2 and Figure 3 show the intensity variation with corresponding pixel distance at different levels (L1 to L6) of the HRA brick 1 and HRA brick 2, respectively. Variation of intensity ratio of the radiographic images indicates the elemental distribution of the contents and the porosity of the sample. This shows that elements are distributed homogeneously at every level compare to one another but each level shows slightly variation of intensity. In case of sample 2 it shows more regular than that of brick-1.

The gray value has been obtained by drawing line profile of 465 × 5 pixel area on the radiographic images of an object. Because of poor irregularity of intensity ratio of each level, little porosity is found in both the case inside the sample.

6.2. Water Penetrating Height and Behavior Measurement at Different Immersion Time

Water penetrating/rising behavior of the RHA brick-1 at different immersion time such as 1, 3, 5, 8 minutes is shown in Figure 4. From this figure it is observed that at 5 to 8 minutes immersion water rises in upward direction almost equally through the all space but at 1 minutes immersion this behavior is slightly different which shows wavy shape. On the other hand in case of RHA brick-2 for 1 minute immersion it shows capillary shape and rests of other immersion time it shows straight line shape, which is shown in Figure 5. The height of water absorption for RHA brick-1 at immersion time 1, 3, 5 and 8 minute is 1.8 - 2.5 cm, 3.2 - 3.4 cm, 3.6 - 3.8 cm (Figure 4) and 4.5 - 4.7 cm, respectively. At the same immersion time for the RHA brick-2 it looks slightly different (Figure 5). From the above investigation it shows that at first 1 minute immersion the water absorption through the RHA brick-1 and brick-2 is very higher than that of 3, 5 and 8 minutes immersion time. From this observation it is pointed out that water penetrating/rising due to 1 and 3 minutes immersion for RHA brick-1 is very poorer than that of the RHA brick-2.

6.3. Determination of Initial Rate of Absorption (IRA)

The absorption rate that water is absorbed by a porous solid is defined as IRA. It is related to the durability, porosity, pore size distribution and water absorption. It is sometimes called rising damp. The quantity, sizes and

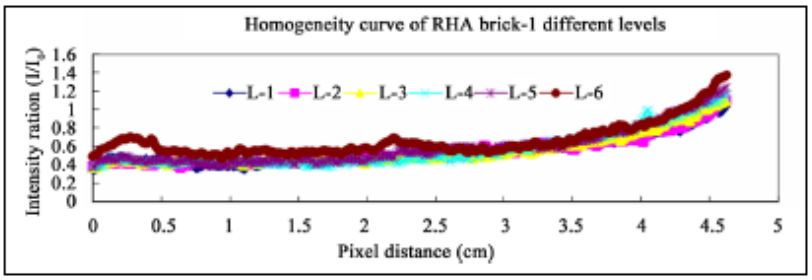

Figure 2. Homogeneity curve of RHA brick-1 at different levels.

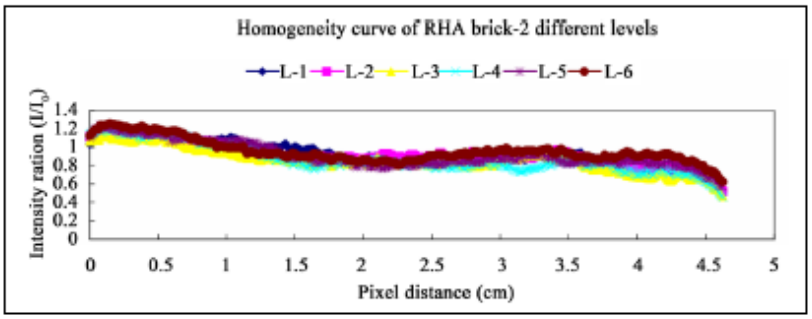

Figure 3. Homogeneity curve of RHA brick-2 at different levels.

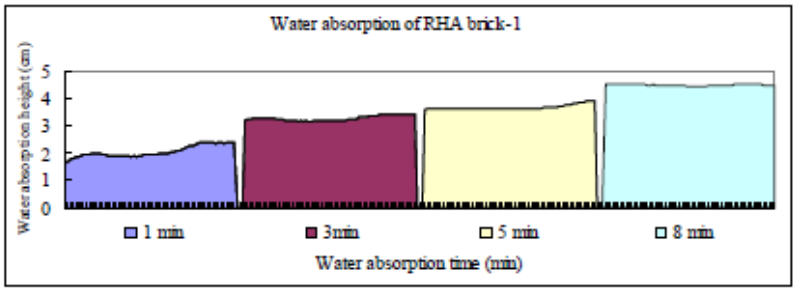

Figure 4. Water absorption/penetration at different immersion time (RHA brick-1).

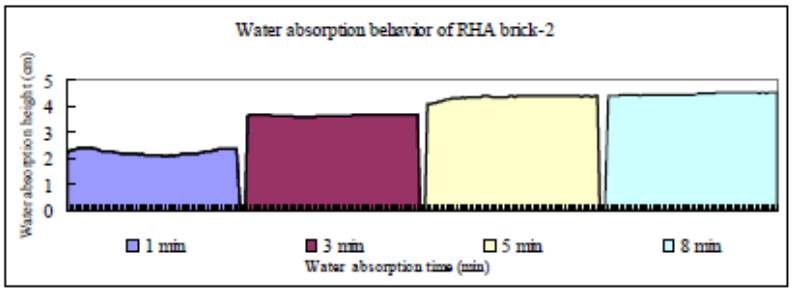

Figure 5. Water absorption/penetration at different immersion time (RHA brick-2).

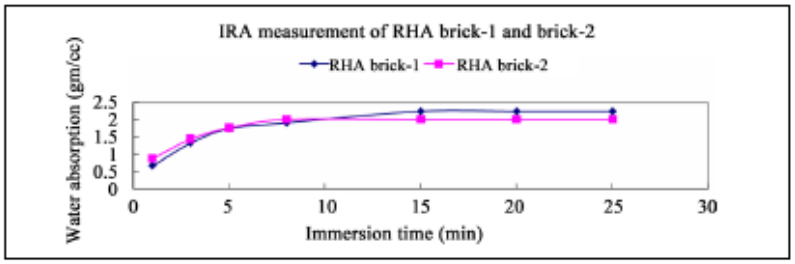

Figure 6. Water absorption/penetration at different immersion time (RHA brick-1 and brick-2).

connection of pores influence the absorption rate of the brick. The IRA is reported in units of gm/(30in^{2}min) [20]. In the present case IRA is measured in units of (gm/cm^{3}min). The results of IRA measurement for RHA brick samples are shown in Figure 6. Initially the rate of water absorption is very fast (1 to 3 minutes immersion) and very similar after this stage (up to 25 minutes water absorption and so on). This investigation is also shows that the water absorption for RHA brick-2 becomes saturated very early than that of the RHA brick-1.

6.4. Observation of Incremental Water Intrusion Area

The incremental intrusion area means the unexpected water absorbed area during immersion period of any respective sample. Figures 7(a)-(d) and Figures 8(a)-(d) show the neutron radiographic images for 1 min., 3 min., 5 min. and 8 min. immersion into water of the RHA brick-1 and RHA brick-2, respectively. The blue straight line is the separator of immersion area and the incremental water immersion area. The lower portion (1 cm × 5 cm)) indicates the immersed area and the upper portion indicates the incremental water intrusion area. Figure 7 and Figure 8 show that growing of incremental intrusion area with immersion time varies very slowly. In that case the water rising due to first 1 min is very faster than that of the next 5 - 8 minutes in both the cases. The relation of incremental intrusion area of the RHA bricks at different immersion time is directly related to the IRA (Figure 6).

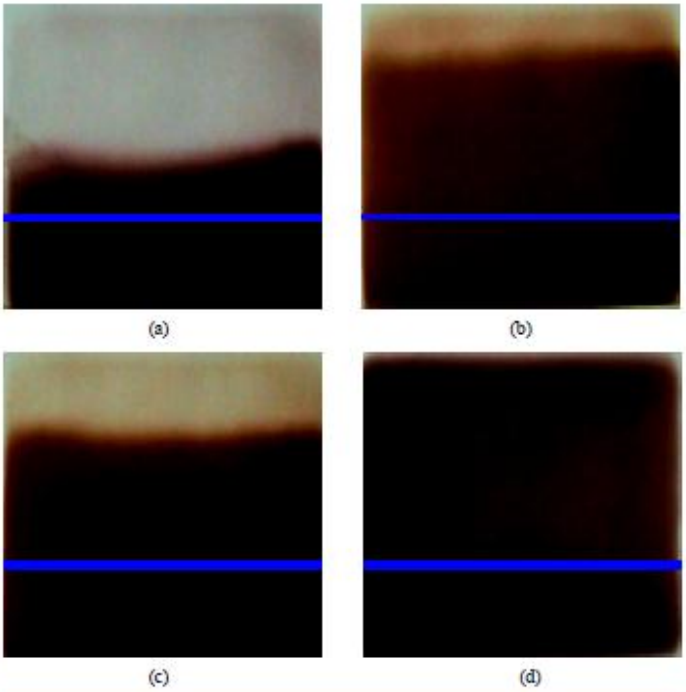

Figure 7. NR images of wet RHA brick-1 at different immersion time. (a) T_w= 1 min; (b) T_w = 5 min; (c) T_w =3 min; (d) T_w = 8 min.

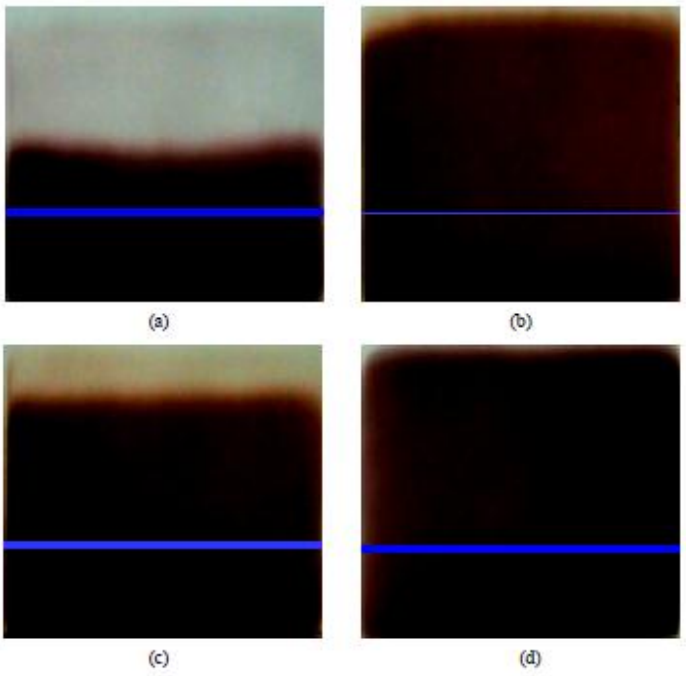

Figure 8. NR images of RHA brick-2 at different immersion time. (a) T_w =1 min; (b) T_w = 5 min; (c) T_w = 3 min; (d) T_w = 8 min.

A typical analysis [21] of rice husk is as ash (22 - 29)%, carbon ≈ 35%, hydrogen (4 - 5)%, oxygen (31 - 37)%, Nitrogen (0.23 - 0.32)%, sulphur (0.04 - 0.08)%. The content of each of them depends on rice variety, soil chemistry, climatic conditions, and even the geographic localization of the culture.

The increasing need for stronger and durable building materials has been to some extent fulfilled by a new concept *i.e.*, blended cement. Blending of reactive rice husk ash in cement has become a common recommendation almost in all the international building codes. Extensive research has been carried out on application of RHA as mineral additive to improve performance of concrete. Reports indicated RHA as a highly reactive pozzolan [22] and is mainly used a replacement of silica fume or as an admixture in manufacturing of low cost concrete block [23] [24]. Other uses of RH are in control of insect pests in stored food stuffs, in the water purification, in vulcanizing rubber, as flue gas desulphurization absorbents. RHA has been found to be effective as an oil spill absorbent, and for use in waterproofing chemicals, flame retardants, and as a carrier for pesticides and insecticides. Its absorbent and insulating properties are useful to many industrial applications. Despite having high potential and suitability in so many well established uses, use of rice husk has been limited. In the competitive market, proper utilization of rice husk and its ash will benefit industrial sectors. The use of rice husk as fuel/electricity generation in efficient manner is likely to transform this agricultural waste material in to a valuable fuel for industrial sectors. A systematic approach to this material can give birth to a new industrial sector of rice husk [23]. It is non-polluting manufacturing process, does not exude gases such as SO_x, NO_x, etc. The use of fly ash and other industrial wastes for making bricks is ecologically advantageous since apart from saving precious top agriculture soil, it meets the social objective of disposing Industrial wastes otherwise are pollutants and nuisance. There are various advantages [25] of RHA bricks in building construction such it can be of good quality with sharp edges, controlled dimensions and offer a plain and even finish. They are resistant to wear and tear which makes them suitable for the internal and external uses. Plastering over brick can be avoided thus achieving further economy; the bonding with mortar and plaster is much greater or better in the case of RHA bricks; this type of bricks can be made in different sizes or shapes, so these can be used in building construction; bulk utilization of RHA helps in solving the pollution problem; these are very easy to produce as they manufacturing, process is simple and machinery required is easily available.

7. Conclusion

RHA brick is very useful brick as a good interior materials of building construction because of its light weight as compare to any other conventional brick, it is easily collected from the rice producing industries with very low price in rice producing countries, elemental distribution is very good at various level, large number of porosity is present with little bit size, and incremental water intrusion area increases very slowly with the increasing immersion time. The information of this study will also be helpful to further study of this type of brick. On the other hand, the NDT technique is the only method to measure the internal voids, cracks, homogeneity and water penetrating through the RHA brick.

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List of Publications of Dr. M. A. Gafur, PSO, PP&PDC, BCSIR

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Original scientific paper

Electrochemical investigation of the corrosion behavior of heat treated Al-6Si-0.5Mg-xCu (x=0, 0.5 and 1) alloys

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Abstract

The corrosion behavior of heat treated Al-6Si-0.5Mg-xCu (x=0, 0.5 and 1 wt %) alloys in 0.1 M NaCl solution was investigated using potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. The potentiodynamic polarization curves reveal that 0.5 wt % Cu and 1 wt % Cu content alloys are less prone to corrosion than the Cu free alloy. The EIS test results also showed that corrosion resistance or charge transfer resistance (R_{ct}) increases with increasing Cu content into Al-6Si-0.5Mg alloy. Maximum charge transfer resistance (R_{ct}) was obtained with the addition of 1 wt % Cu and minimum R_{ct} value was for Cu free Al-6Si-0.5Mg alloy. Due to addition of Cu and thermal modification, the magnitude of open circuit potential (OCP), corrosion potential (E_{corr}) and pitting corrosion potential (E_{pit}) of Al-6Si-0.5Mg alloy in NaCl solution were shifted to the more noble direction.

Keywords

Al alloy; Nyquist plot; corrosion rate; tafel plot; EIS

Introduction

Aluminium and its alloys are considered to be highly corrosion resistant under the majority of service conditions [1]. The various grades of pure aluminum are the most resistant, followed closely by the Al-Mg and Al-Mn alloys. Next in order are Al-Mg-Si and Al-Si alloys. The alloys containing copper are the least resistant to corrosion [2]; but this can be improved by coating each side of the copper containing alloy with a thin layer of high purity aluminium, thus gaining a three

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Sintering Characteristics of La/Nd doped $Bi_4Ti_3O_{12}$ Bismuth Titanate Ceramics

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Abstract:

A good understanding about the properties of La/Nd doped Bismuth Titanate (BIT) ceramics at high temperature is very important as the new materials being developed based on the BIT. Pure BIT, La doped (BLT), Nd doped (BNT) and La and Nd co-doped BIT (BLNT) powders were synthesized by solid state reaction method. Prepared powders were calcined at different temperatures and structural properties measured by XRD. For pure BIT better crystal quality was obtained at 750 ^{0}C and for both BLT and BNT better result obtained at 800 ^{0}C. Calcined powders were formed into pellets and sintered at different temperatures and its dielectric properties were characterized. Optimum sintering temperature for both BLT and BNT showed was 850 ^{0}C and La and Nd co-doped bismuth titanate (BLNT) revealed optimum sintering temperature of 950 ^{0}C. Therefore, optimum sintering temperature of bismuth titanate was increased due to La and Nd doping.

Keywords: *Bismuth Titanate, La/Nd doping, Sintering, XRD, Dielectric properties.*

1. Introduction

Bismuth Titanate ($Bi_4Ti_3O_{12}$) (BIT) is an attractive material for their lead free character with high curie temperature, high dielectric constant, high remnant polarization, excellent piezoelectric and electro-optical properties, relatively low possessing temperature and high breakdown strength and have become a potential candidates for the piezoelectric applications (transducers and actuators, ultrasonic devices, medical imaging detectors), Non volatile memory storage, and optical displays[1,2]. However, BIT suffers from not enough remnant polarization($2P_r$), high coercive field ($2E_c$) and high leakage current and domain pinning due to defects such as Bi vacancies accompanied by oxygen vacancies which is an obstacle for industrial applications. A large number of researches have been performed dynamically in both thin film and bulk ceramics and studies are still in steps forwarded to overcome the hitch of BIT.

It is well known that, ion doping by suitable cations (La, Nd, Pr etc.) is a best approach for the improvement of ferroelectric performance of BIT [3-6]. Among these cations, lanthanide elements, Nd [r(Nd^{3+})=1.27Å] and La [r(La^{3+})=1.36Å] have been recognized as the materials to substitute Bi [r(Bi^{3+})=1.40Å] in the A-site of perovskite structure. The radii size of Bi^{3+} ion is compatible with that of Nd^{3+} and La^{3+} ions, suggested that both materials can eagerly for this substitution in perovskite bismuth titanate. Recently La-substituted BIT having composition of ($Bi_{3.15}La_{0.75}Ti_3O_{12}$) (BLT) is regarded as one of

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Mechanical properties of Gelatin –Hydroxyapatite composite for bone tissue engineering

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Abstract

In this study, hydroxyapatite (HAp) and gelatin (GEL) scaffolds were prepared to mimic the mineral and organic component of natural bone. The raw material was first compounded and resulting composite were molded into the petridishes. Using Solvent casting process, it is possible to produce scaffolds with mechanical and structural properties close to natural trabecular bone.The mechanical properties of composites were investigated by Thermo-mechanical analyzer (TMA), Vickers microhardness tester, Universal testing machine. It was observed that the composite has maximum tensile strength of 37.13MPa (oven drying) and % elongation of 7.68 (Oven drying) and 2.04 (Natural drying) at 15% of Hap respectively. These results demonstrate that the prepared composite scaffold is a potential candidate for bone tissue engineering.

Keywords: Hydroxiapatite; Thermomechanical analysis; Tensile strength; Tensile elongation

Introduction

Biomaterials play a very important role in tissue engineering, which is aimed to provide replacements for tissues and organs which have been damaged or lost as a consequence of disease, aging or accident (Safinya *et al.*, 1996). A part of biomaterials is designed for replacing bone and/or inducing bone formation. From the biological perspective the natural bone matrix is a combination of organic/inorganic composite materials and consists of a naturally occuring polymer (collagen) and a biological mineral (apatite) (Cho and Lin, 2008). Further blending with inorganic materials can modify the mechanical properties as well as the degradation rates of the materials. The designed composite scaffold should combine the advantages of both components. Moreover, the natural composite material should have an excellent balance between strength and toughness, both of which should be superior to those of the individual components (Ma, 2008). Therefore, instead of using a single material type for synthesis it is a natural strategy to combine polymers and inorganic ceramic i.e. hydroxyapatite (HAp) to fabricate scaffolds that meet all the requirements desired for particular applications in tissue engineering. Thus polymer/inorganic composite scaffolds have attracted the attention of researchers.

Over the past decades, growing interest has been focused on the development of inorganic–organic hybrid biomaterials in order to mimic natural materials' composition and structure. A minority component of organic macromolecules in ceramics such as proteins or polymers are usually used to control nucleation and growth of inorganic crystals, and thus to improve microstructure and physical properties. This related methodology has a great potential for the design and engineering of novel biomaterials with special functionalities (Safinya *et al.* 1996).

According to a literature review the compressive strengths of cortical and cancellous bone are in the ranges 100–230 and 2–12 MPa, whereas the Young's moduli are in the ranges 7–30 and 0.5–0.005 GPa, respectively (Hutmacher *et al.*, 2007). Among the commonly used polymers, poly (lactic acid) degrades within the human body to form lactic acid, a naturally occurring substance which is easily removed from the body material. One of the main disadvantages of biodegradable polymers used in tissue engineering is their poor mechanical properties. Thus, there have been many studies to improve the mechanical properties of biodegradable polymers by incorporation of other materials (Goutard *et al.*, 1993). In biomineralized tissues as bone, teeth and calcified tendons, the collagen matrix is stiffened by the apatitic crystals which act as filler particles (Vincent *et al.*, 1990). Due to the close structural relationship between the two components, the resulting composite material exhibits

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Characterization of Scaffold Prepared by Blending Nanobioactive Glass and Graphene Oxide-Gelatin Hydrogel Solutions for Bone Tissue Engineering

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A biologically promising material with improved mechanical property has been developed successfully. In this study graphene oxide of varying amount were incorporated into a gelatin-nanobioactive glass (nBG-Gel) matrix through a freeze drying technique. The compressive strength test was carried out to determine the reinforcement effect of GO and it demonstrated that nBG-Gel matrix with GO shows a remarkably higher compressive strength and a compressive modulus than those of the bare. The porous structure (in terms of pore size and pore interconnectivity) of the composite scaffolds were examined. Morphological characterization revealed the formation of an interconnected porous hybrid of size range of 100 μm to 600 μm. The thermal properties of the hybrid were analyzed using Simultaneous Thermal Analyzer (STA). All the experimental results demonstrate that the prepared hybrid scaffold is a potential candidate for bone tissues engineering.

Keywords:

RESEARCH ARTICLE

1. INTRODUCTION

Tissue engineering presents an alternative approach for autografts in healing of diseased, damaged and traumatized bone tissue.[1] A tissue engineering approach compromise of controling tissue formation in three dimensions (3D), a considerable porous scaffold is crucial for nutrition and metabolite diffusion. In addition to the processed 3D geometry for the tissue to be engineered, the scaffold should provide the microenvironment (synthetic temporary extracellular matrix) for regenerative cells, supporting cell attachment, proliferation, differentiation, and neo tissue genesis.[2] Because bone consists of a porous composite of interpenetrating phases of hydroxyapatite and collagen, the scaffolds for bone regeneration should be similarly porous composites with interpenetrating ceramic and polymer phases. While scaffolds are expected to disappear after implantation *in vivo*, a certain level of mechanical strength is also required for the scaffolds to withstand a certain level of physiological loading.

Gelatin, is a partial derivative of collagen, and has a wide range of uses in the pharmaceutical drug delivery and other biomedical applications.[3] Regarding bone tissue engineering, gelatin was used as a polymer to increase the flexibility of the porous structure.[4,5] However, it has a poor mechanical property[3,6] and requires quantities that exceed the physiological dose. Bioceramics are often used in combination with biodegradable polymers such as gelatin to achieve the best possible mechanical and biological performance. The characteristic of bioactive glasses (BG) include; excellent bioactivity, ability to deliver cells, and controllable biodegradability[7] of other bioceramics of concern. Recently bioactive glasses been reported to be one of the possible most exciting future clinically applicable material to fabricate optimal scaffolds with osteogenic and angiogenic potential.[8] Bioactive glass scaffolds alone are quite brittle and not easy to handle because of highly interconnected pores, the mechanical strength of the prepared porous scaffolds is low.[9–11] On the other hand, scaffolds based upon polymeric material alone such as gelatin are not ideal in terms of their mechanical strength.[12] To address these issues and to keep a proper balance between the biological and the mechanical strength, the addition of graphene oxide fillers in nano bioglass-gelatin matrix been used.

Graphene, is a potential biomaterial because of its large surface area, nontoxicity, high dispersibility and

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Chemical Characteristics of Hydroxyapatite from Oyster Shell by Thermo-Chemical Process

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ABSTRACT: Oyster shell is basically made of calcium carbonate. An attempt is made to convert calcium carbonate of oyster shell to hydroxyapatite (HA), a substitute material for bone and teeth in orthopedic and dentistry field due to their chemical and biological similarity to human hard tissue. In the present work, hydroxyapatite (HA) was successfully synthesized by wet precipitation method using Ca $(NO_3)_2$, and $(NH_4)_2HPO_4$, and NH_4OH as starting materials. The oyster shell was found to decompose within 1000^0C to all the carbonate phases. The calcined oyster shells were then treated with acids followed by different chemicals in ammoniacal media maintaining proper stoichiometry to produce fine hydroxyapatite (HA) as filter cake with a Ca/P molar ratio of 1.67. The dried HA powder was extremely pure. Different characterization techniques were adopted both for calcined oyster shell and HA by scanning electron microscopy (SEM), X-ray diffractometer (XRD), fourier transform infrared spectroscopy (FTIR) and thermo gravimetric analyzer (TGA). The absorption bands corresponding to phosphate and hydroxyl functional group which are characteristics of hydroxyapatite were confirmed by FTIR. Besides its effectiveness in bone substitution, the prepared HA holds great potential in the field of dental application.

KEYWORDS: Oyster shell; Hydroxyapatite; Wet precipitation; Sintering; Calcination.

I. INTRODUCTION

Materials which are used for the repair and reconstruction of diseased or damaged parts of the musco-skeletal system are defined as biomaterials such as hydroxyapatite (HA), bioglass, biopolymer, and Ca-phosphates etc (Burg *et al.*, 2000). A lot of research has been carried out to find out the substitution to support the bone in the medical field. Presently steel as support to bones is used but it can react with body fluids [T = 37^0C and pH = 7.4] when kept for many years, so it requires substitute. Next comes to the inert materials like bioglass but due to formation of fibrous tissue of variable thickness they can lead to tumor. Now-a-days ceramics specially the bio ceramics are the better alternatives since they have high corrosion resistance (Urist and Johnson, 1941), better compressive strength, relatively low density and low weight. Porous bio-active ceramics such as hydroxyapatite, Ca-phosphates are attractive for bone regeneration and reconstruction due to their bone bonding ability and good growth property (Sevitt, 1981). Among them hydroxyapatite is taken as the best alternative as it contains same chemical nature and similar crystallographic structure with bone excellent biocompatibility (McKibbin *et al.*, 1970).

Hydroxyapatite (HA) $Ca_{10}(PO_4)_6(OH)_2$ is a synthetic biomaterial. Due to its chemical and structural similarity with the mineral phase of bone and teeth, HA is widely used for hard tissues repair. As a result, this inorganic phosphate has been studied extensively for medical applications in the form of powders, composites or even coatings (Weng *et al.*, 2000; Cheng *et al.*, 2003; Riman *et al.*, 2002; Huang *et al.*, 2000; Weng *et al.*, 2003; Weng *et al.*, 2002; Choi *et al.*, 2004; Weng and Baptista, 1998; Cheng *et al.*, 2003; Cheng *et al.*, 2001; Weng *et al.*, 2002). It is also observed that dense sintered HA has many bone replacement applications and is used for repairing bone defects in dental and orthopedic sites, immediate tooth replacement, augmentation of alveolar ridges, pulp capping material and maxillo facial reconstruction, etc (Shiny *et al.*, 2000). For substituting or repairing the bone, the designed material must has the ability to create a bond with the host living bone (Kokubo *et al.*, 2003). Hence, it is always desirable to include a high degree of crystallinity and chemical stability among the desirable properties of an ideal hydroxyapatite (Jha *et al.*, 1997; Tsui *et al.*, 1998).

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Impact of doping on Structural, Electronic and Optical Properties of Cobalt Ferrite Prepared by Solid-state Reaction

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Abstract— Cobalt ferrite ($CoFe_2O_4$) is a promising material due to its exceptional ferrelectric, optical, electronic and magnetic properties that are most important for device applications. Partial substitution of iron with transition metals might play an important role to improve its different properties useful in various applications. Degree of crystallinity, particle size, electrical conductivity, optical absorption and low temperature magnetization of cobalt ferrite were measured before and after doping with transition metals Zr, Zn and Cd. The crystallinity improved for doping effect. The charge capacity and optical absorptivity also reported to be increase for doping effects.

Keywords— Calcinations, Characterization, Solid-State reaction, Ferroelectric materials, X-ray diffraction.

I. INTRODUCTION

Cobalt ferrite ($CoFe_2O_4$) are well-known ferroelectric materials. Ferroelectric magnets simultaneously show th properties of ferroelectrics and ferromagnets [1]. Ferroelectric materials possess two or more orientational states in th absence of an electric field and can be changed from one to another of these states by the application of an electric field. Th combination of magnetic and electrical properties makes ferroelectric-magnet useful in many technological applications. I has covered a wide range of applications including electronic devices, high density information storage devices, high rate o change of strain with magnetic field, highcoercivity, moderate saturation magnetization, high Curie temperature T_C, photo magnetism, good electrical insulation etc. [2]-[6]. Cobalt ferrites are suitable for magnetic recording applications such a audio and videotape and high-density digital recording disks etc. [7]. Cobalt ferrites are the subject of much interest due t their unusual optical, electronic and magnetic properties. Substitution of rare earth (Y^{3+}, Gd^{3+}, Ho^{3+}, Sm^{3+}, Nd^{3+}) ion int cobalt ferrites has been reported to lead to structural distortion that induces strains in the material which indicate improve crystallinity of the sample and to affect the electrical and magnetic properties significantly. The rare earth doped cobal ferrites found an application in high frequency devices and power supply due to high resistivity and low dielectric losses [8] [9]. Cobalt ferrite has become an ingredient in the formation of metallic/magnetic nano-composites due to the observation of a wide range of magnetic, electric and other novel properties in its derivatives [10],[11]. Some transition metals wer doped in iron site of the cobalt ferrite [$CoFe_{2-x}M_xO_4$, M=Zn, Zr, Cd] to enhance its structural, electronic, dielectric, optica and magnetic properties that might play important role in high efficiency device applications.

II. EXPERIMENT

High-purity (>99.9%) powders of CoO and Fe_2O_3 were used as the starting material for preparation of $CoFe_2O_4$ Initially we studied the phase formation employing a thermo balance (TG/DTA 630) in order to establish the procedure fo synthesizing $CoFe_2O_4$. A stoichiometric mixture of CoO and $Fe_2O_3$with molar ratio of Co:Fe = 1:1, was heated in a st atmosphere with a heating program as shown in the inset of Fig.1. A representative thermo gravimetric (TG) curve obtaine for the raw material mixture is shown in Fig.1. From this TG analysis, it was found that the weight loss below 600°C is too large.

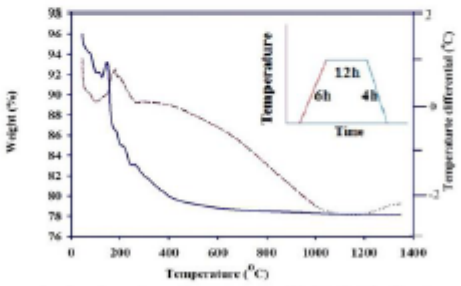

Fig.1 Thermo gravimetric curve for the phase formation process of $CoFe_2O_4$ The inset represents the heating program employed.

Optical, Thermal and Electrical Properties of Jute and Glass Fiber Reinforced LDPE Composites

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Abstract

ıte and glass fiber reinforced low density polyethylene (LDPE) composites were prepared sing compression molding technique at 120° C with various percentage of fiber content. hermal, optical and electrical properties of both composites were studied in this article. hermal analysis of the composites confirmed the better thermal stability of glass fiber LDPE ɔmposites than that of the jute composite. Superposition of the absorption of LDPE and lass fiber in the composites has been confirmed by the peak and line shape of absorptions. he absorption peaks also indicates a better conjugation between the elements of composites. lectrical studies suggest that for both composite capacitance decreases with increase in equency and voltage, which suggests good electrical properties of LDPE based composites.

ey Words: Fiber content, thermal stability, absorption peak, electrical properties, ɔmposites.

ıtroduction

ven though there are lots of conventional materials such as metal alloys, ceramics, and olymeric materials that are widely used in various branches of modern technology, however, searches on the development of new materials have never been stopped. Now a day, many f our modern technologies require materials with unusual combinations of properties that ınnot be meet by conventional materials. In this perspective it can mentioned that aircraft

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SAGE

Study of the electric properties of palm fiber-reinforced acrylonitrile butadiene styrene composites

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Abstract

The electrical properties like AC conductivity, AC resistivity, and dielectric constant of the palm fiber-reinforced acrylonitrile butadiene styrene composites were investigated using impedance analyzer at room and variable temperatures. Palm fiber was collected from 10 trees of different age group from Comilla region in Bangladesh. Three sets of samples were prepared for three different wt (%) (5%, 10%, and 20%) of fiber contents using Injection Moulding Machine. Each set has 10 composite samples in which first five of them are made with 11 to 20 years-aged palm fiber and latter five of them are made with 5 to 10 years-aged palm fiber. The AC electric conductivity increases with the increase of frequency, wt (%) of palm fiber in the PF-ABS composites, and with the increase of temperature. The AC resistivity decreases with the increase of frequency, with the wt (%) of fiber content in the PF-ABS composites and with the increase of temperature. Moreover, the comparison between the PF-ABS composites with 11 to 20 years-aged palm tree shows better result than 5 to 10 years-aged palm tree. The dielectric constant of the composites decreases with the increase of frequency. With the addition of palm fiber content in composites, dielectric constant increases. The dielectric constant also increased with the increase of temperature.

Keywords

Conductivity, resistivity, dielectric constant, polarization, polar group

Introduction

Composite materials reinforced with cellulosic fibers have received interest in recent years and are widely using in many fields such as civil, industrial, military, space craft, and biomedical application mainly because of their excellent thermo mechanical properties, biodegradability, low density, and non-toxicity.[1–9]

It is well known that composites can be produced exhibiting enhanced properties that the constituent materials may not exhibit. For instance, from the combination of different number or fillers with polymer matrices one can produce polymer-matrix composites, material important to the electronic industry for its dielectric properties in the use of capacitors. The effective utilization of filled polymers depends strongly on the ability to disperse the filler homogeneously throughout the matrix. The interface properties also strongly affect the characteristics and performance of

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Influence of Talc Filler Content on the Mechanical and DC Electrical Behavior of Compression Molded Isotactic Polypropylene Composites

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Abstract Isotactic polypropylene(iPP) and iPP-talc composites with compositions of 9:1, 8:2, 7:3, 6:4, 5:5 wt. ratio were prepared by extrusion cum compression molding methods. Various mechanical properties such as Young modulus (YM), tensile strength (TS), elongation- at -break (EB%), flexural strength (FS), flexural modulus (FM) and electrical behavior of these composites were investigated. YM of iPP-talc composites increases with the increase of talc content whereas TS and EB (%) of the composites decrease with talc content. The YM value of the composites with 30 wt% talc is the highest. The FS increases with the addition of talc up to 30 wt% and above this it decreases. But flexural break (%) decreases with the increase of talc content. FM increases slowly up to 20 wt% talc and then it increases rapidly for 30 wt% talc. The current-voltage (I-V) characteristics of the composites were recorded in the voltage range from 0 to 120 V DC at different temperatures. It is evident that I-V curves show ohmic behavior in the lower voltage region and in the higher voltage region the contact is non ohmic, which suggests that the current may be due to space charge limited conduction (SCLC) for iPP and Schottky or Poole–Frenkel (PF) conduction mechanisms in iPP-talc composites. Electrical conductivity is found to increase with increasing temperature but it decreases with the increase of talc content for these composites. The activation energy of all the samples has higher value in high temperature region than those at low temperature region.

Keywords Isotactic polypropylene – talc composites, Mechanical properties, Electrical behavior

1. Introduction

In comparison with metals, polymers can offer better processing, a lower density, a higher strength-to weight ratio, better resistance to corrosion, and often a better price/performance ratio. A current increased demand for applications of synthetic polymers in the automotive and aeronautic industries is evident, mainly in the utilization of polyethylene, polypropylene (PP), polycarbonate, and polyamide components in the interior, exterior, and other functional parts of the vehicle [1].

Isotactic polypropylene (iPP) is becoming one of the most important commodity polymers, widely used in technical appli cations. Because of its intrinsic properties, such as a high melting temperature, low density, high chemical inertness and the capability of being toughened with elastomers, iPP has found a wide range of applications in the food packaging, electrical, and automotive industries. Moreover, iPP is one of the most favorable matrices for high-volume, low-cost composites and blends [2]. Its applications are greatly extended by adding inorganic fillers such as talc, calcium carbonate, mica, glass, etc to improve mechanical properties, thermal resistance, and dimensional stability, all at a low cost [3].

This study is focused to reinforcement of iPP with the addition of the mineral talc. Talc is a hydrated magnesium silicatemineral having the chemical formula [$Mg_3Si_4O_{10}(OH)_2$], widely used in polymers as a reinforcing filler. Talc contains 31.7% MgO, 63.5% SiO_2 and 4.8% H_2O. Talc deposits are probably formed by hydrothermal attention or contact meta morphism of preexisting rocks. Its plate-like structure provide the talc filled materials with tailored properties to be used in some industrial and commercial applications such as in refrigerators jackets, packaged components, blocking of infrared radiation in agricultural films, and in automotive and appliance markets. Talc filled iPP composite has low specific gravity and combines excellent chemical resistance with low cost [4]. Researchers around the world work to develop both new composite materials and also improve existing ones. A large no of research works has been dedicated to improve the properties

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Characterization of Isotactic Polypropylene/ Talc Composites Prepared by Extrusion Cum Compression Molding Technique

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Abstract

Extrusion-Compression molded isotactic polypropylene (iPP) composites containing 10 wt%, 20 wt%, 30 wt%, 40 wt% and 50 wt% of talc filler were studied by scanning electron microscopy (SEM), simultaneous thermal analysis (STA) and physical testing. The scanning electron microscope (SEM) micrographs of neat iPP and composites with 10 wt%, 20 wt%, 30 wt%, 40 wt% and 50 wt% talc content show that neat PP, 10 wt%, 20 wt%, and 30wt% talc composites surface is smooth in comparison to 40 wt% and 50 wt% talc composites. It is also observed that talc is dispersed uniformly in the matrix and this uniform dispersion is not decreased even with talc content as high as 30 wt% talc. The composites of 40 wt% and 50 wt% talc contain more crack, agglomerates or larger particles. Bulk density of the composites decreases with the increase of talc content. With the increase of percentage of talc and period of immersion, the water absorption (WA) increases. Thermal analyses indicate a considerable increase of thermal stability of the composites with filler addition.

Keywords

Isotactic Polypropylene, Water Absorption, Thermal Behavior, Talcs

1. Introduction

Fillers-reinforced polymer composites have attracted much attention to the researchers due to the fact that inclusion

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Mechanical and Spectroscopic Properties of Rice Husk Reinforced Polypropylene Composites: Effect of Sodium Hydroxide

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Abstract Rice husk reinforced polypropylene-based composites were prepared by conventional compression molding. Composites contained 5-20% (by weight) of rice husk as the reinforcing agent. Then mechanical and spectroscopic properties of the composites were evaluated. Rice husk was also treated with sodium hydroxide (NaOH) solution (1% by weight) to investigate the effect of NaOH on the mechanical properties of the fabricated composites. Loading of rice husk revealed that mechanical properties of polypropylene based composites significantly improved. Fourier transform infrared (FTIR) spectroscopy confirmed the existence of hemicelluloses and lignin in the neat composite. Interfacial properties were investigated by scanning electron microscopy (SEM).

Keywords Composites, Polypropylene, Rice husk, Mechanical properties, SEM

1. Introduction

Composite materials are widely using in civil construction, automobiles, space and aircraft applications, naval manufacturing and many others. Composite materials are manufactured by combining two dissimilar materials into a new material which is better suited for a particular application than either of the original material alone. Nowadays, modern technologies require materials with unusual combination of properties that cannot be met by the conventional materials. A relatively new class of composite material isfiber reinforced polymers which is manufactured using fiber and polymer. Composite materials are efficient and economical for use in a variety of engineering applications. There are two types of matrix materials in fiber reinforced polymer based composites, i.e., (a) thermo-plastics and (b) thermo-sets. While different types of fibers are using as reinforcing filler in the composites. It is to be noted here that thermoplastic resins achieved much interest due to their economic and mechanical advantages such as easy fabrication, intrinsic recyclability, unlimited shelf life, high toughness and good moisture resistance. Fibers provide stiffness and tensile capacity to the composites and improve their mechanical characteristics [1-3].

Every year billion of tons of agricultural crop residues are produced around the world. Among this large amount of residues, only a small quantity of them are utilized as household fuel or fertilizer and the rest is burned in the field. As a result, it causes air pollution. Utilization of the agricultural crop residues as reinforcement of polymer composites can solve this problem. Application of natural fibers as reinforcing agent to composites gained much interests due to increased environmental awareness and consciousness throughout the world. Natural fibers are now considered as an alternative to synthetic fibers for use in various fields such as building and construction, packaging, automobile and railway coach interiors and storage devices, and various load bearing applications. They are environmentally friendly, biodegradable, widely available, renewable, cheap, and have low density. The biodegradability of plant fibers contribute to a healthy ecosystem and their high performance satisfies industrial economy. Plant fibers are light in weight compared to glass, carbon, and aramid fibers. Recently, a wide variety of natural fibers have been investigated for the reinforcement of the composites. These include jute, wood, rice husk, banana fiber, sisal, bamboo, coir, hemp, flax, kenaf, and ramie [4-7].

Composite materials prepared from unmodified plant fibersoften exhibit unsatisfactory mechanical properties. To overcome this drawbacks, surface treatments are required prior to composite fabrication. The mechanical properties of

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Study of Different Chemical Treatments for the Suitability of Banana (*Musa oranta*) Fiber in Composite Materials

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Abstract—The present works demonstrate banana fibers which were chemically modified with 7.5% NaOH & 0.055% $KMnO_4$. The effects of modification were determined by measuring various physical and mechanical properties such as moisture absorbance, tensile strength, % elongation break and characterized by various methods such as SEM, IR etc. The ultimate tensile strength of treated fibers was lower than that of raw fibers. Percentage elongation and moisture absorption increase on treatment.

Keywords— Banana fiber (Natural filler), Chemical treatment, Mechanical properties, Physical properties, Polypropylene (Matrix), Polymer composites

1 Introduction

Fibers are used in composite materials as reinforcing constituents. Early in the development of composites, the only reinforcements available were derived from traditional textiles and fabrics. Particularly in the case of glass fibers, experience shows that the chemical surface treatments or "sizing" required processing these materials into fabrics and other sheet goods were detrimental to the adhesion of composite polymer to the fiber surface.[1] Techniques to remove these materials were developed. Simultaneously natural fibers were considered as the substitution for glass and carbon fiber in polymer composites. Their potentiality for being used in molded article, not needing high strength for acceptable performance, has been tried in equipment housing, roofing, low cost housing, large diameter piping, and many other purposes. Among all natural fibers jute is the most frequently used fiber [2]. Natural fibers are naturally obtained from different types of plants. Natural fibers can be divided into three groups, i.e. vegetable fibers (flax, hemp, sisal, etc.), protein fibers (wool, silk, chitin, etc.), and mineral fibers (asbestos, etc.). Vegetable fibers are renewable with good mechanical properties which justify their use as reinforcement for polymers. They are now being used or expected to be used extensively for composite materials due to the following advantages [3]. Low specific weight, which results in a higher specific strength and stiffness than glass, is a benefit especially in parts designed for bending stiffness. It is a renewable resource, the production requires little energy, and CO_2 is used while oxygen is given back to the environment. It is produced with low investment at low cost, which makes the material an interesting, predicable for low wage countries. Ethnically processed, no wear of tooling, no skin irritation, and thermal recycling are possible, where glass causes problem in combustion furnaces. They have good thermal and acoustic insulating properties [4, 5, 6]. In spite of having a lot of advantages, natural fibers have some disadvantages such as lower strength properties particularly its impact strength, variable quality which depends on unpredictable influences such as weather and high moisture absorption which eases swelling of the fibers, low processing temperature, and low durability which can be improved by fiber treatment considerably.

Banana is the common name for a type of herb and also the name for the herbaceous plants that grow this herb. These plants belong to the genus Musa. They are native to the tropical region of Southeast Asia. There are about 100 different species of banana. In general, banana is of the leaf fibers that are coarser than the baste fibers [7]. Applications are ropes and coarse textiles. Within the total production of leaf fibers, sisal is the most important. The stiffness is relatively high and it is often applied as binder twines. In some interior application sisal is preferred because of its low level of smell compared to fiber like flax which at increased temperatures (NMT) during manufacturing process can cause smell [8].

Since the fiber reinforced composite material relies on the fiber for its strength and stiffness, it is essential that this constituent passes high strength and high modulus compared to the matrix, then with the appropriate volume fraction accompanied by control of fiber orientation and fiber dimensions the mechanical behavior such as strength, toughness and stiffness can be optimized. Variability in strength of fiber is also a con-

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Hybrid Natural Fibers/Isotactic Polypropylene Composites with Degraded Polypropylene as Compatibilizer

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Abstract

Interest on the field of natural fiber-thermoplastic composite has been considerably increased because of the ne environmental legislation to use biodegradable fibers instead of pollution causing synthetic fibers like carbon, glass etc. In thi investigation, an attempt was taken to use of inexpensive areka palm leaf fibers (APLF) as reinforcement of polypropylen (PP) composites. Degraded polypropylene (DgPP) was added in composites as a compatibilizer of hydrophilic natural fiber an hydrophobic PP matrix. The mechanical properties (tensile and flexural) of composites were more pronounced with th addition of 5 wt% DgPP. The weight percentage of APLF and PP were varied to get better mechanical strengths of composite The (tensile and flexural) strengths were increased upto 10 wt% fiber loadings and thereafter decreased whereas the (tensil and flexural) modulus were increased with the increases of fiber loading upto maximum (20 wt%). Hybrid fibers composite were also fabricated with different combinations of APLF, pineapple leaf fiber (PALF), DgPP and PP (5+5+5+85 wt% an 5+10+5+80 wt%). The hybrid fiber composite with (5+5+5+85) wt% combination was exhibited superior mechanica properties than unreinforced PP and other composites. The water absorption properties of the composites were also studied.

Keywords

Areka Palm Leaf Fiber, Polypropylene, Hybrid Natural Fiber Composites, Mechanical Properties, Water Absorption

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1. Introduction

To protect the earth from 'white pollution', there is no alternative to use bio-based materials rather than synthetics. Natural fibers are the major contributors of daily needed bio-based materials. They have wide range of applications in textile, construction, paper, cosmetics, pharmaceutical, medical, automobile etc. Newly discovered composite technology is extended their use in different industrial applications. The conventional natural fibers such as jute, coir, sisal, hemp, wood flour etc. has been used for the manufacture of composite materials from last three decades [1-4]. Recently, some non-conventional agro-residual fiber e.g. okra, banana, pineapple leaf, corn husk, areka palm lea etc. have attracted much attention as a filler of polymer base composite materials for their low cost, high modulus an renewability [5-9]. The reinforcement of those natural fiber is considerably improved the mechanical properties o polymer composites. However, such mechanical properties o natural fiber composites are much lower than glass or othe high performance fibers composites. The coarseness, voi content, cluster formation and low interface properties are th barriers of the proper distribution of fiber in composites an hence showing ruthless performance. However, th combination of two or more natural fibers can improve th

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Study on Physio-Mechanical Properties of Rice Husk Ash Polyeste Resin Composite

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Keywords: Rice husk ash, polyester resin, bulk density, flexure strength and elastic deformatio

ABSTRACT

The rice husk ash/polyester resin composites were prepared by compression molding meth and their physical and mechanical properties were studied by universal testing machine. T hardness of the composites were tested by Leeb rebound hardness tester and Vickers hardness tes .The bulk density of the rice husk ash/polyester resin composite decreased with the addition of rice husk ash, and the water absorption also found to be increased with increase in soaking tir Flexure strength of the composite was decreased randomly with an increase in rice husk ash conte The elastic modulus for the flexure strength increased up to the percentage 0-10% but decreased 15% and 20% of the rice husk ash/polyester composite. The compressive strength of the composi was decreased randomly with the addition of rice husk ash content, and the elastic modulus compressive test was increased firstly on the addition of rice husk ash, but it was decreased a 5%. The Hardness of the prepared composite was found to be decreased with an increase of addition of rice husk ash content due to elastic deformation.

1. INTRODUCTION

Composites, the wonder material with light-weight; high strength-to-weight ratio and stiffn properties have come a long way in replacing the conventional materials like metals, wood etc. unique feature of composites is that the characteristics of the finished product can be tailored t specific engineering requirement by the careful selection of matrix and the reinforcement type. examples aircraft engineers are increasingly searching for structural materials that have densities, are strong, abrasion and impact resistant, and are not easily corroded. Frequently, stro materials are relatively dense, also increasing the strength or stiffness generally result in a decre in impact strength [1-4]. Nature is full of examples wherein the idea of composite materials is us The coconut palm leaf, for example, is nothing but a cantilever using the concept of fi reinforcement. Wood is a fibrous composite; cellulose fibers in a lignin matrix. The cellulose fib have high tensile strength but are very flexible (i.e. low stiffness), while the lignin matrix joins fibers and furnishes the stiffness. Bone is yet another example of a natural composite that suppo the weight of various members of the body [5-7]. Composites are combination of two materials which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or partic and are embedded in the other materials called the matrix phase. The reinforcing materials and matrix material can be metal, ceramic, or polymer. Typically reinforcing materials are strong w low densities while the matrix is usually a ductile, or tough, material [8-10]. If the composite designed and fabricated correctly, it combines the strength of the reinforcement with toughness

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Preparation and Characterization of Copper Oxide Nanoparticles Synthesized via Chemical Precipitation Method

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Abstract

Copper oxide nanoparticles (CuO-NPs) were synthesized via chemical precipitation method using copper (II) chloride dihydrate and sodium hydroxide. Then nanoparticles were characterized by using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDX), and Fourier Transform Infra Red (FTIR) spectroscopy. The XRD patterns and EDX spectra showed that the prepared CuO-NPs were highly pure, crystalline and nano-sized. The SEM image suggested that nano particles were spherical and there was a tendency of agglomerations. The nanoparticles showed interactions between copper and oxygen atoms supported by FTIR studies.

Keywords

Copper Oxide, Nano Particles, Chemical Precipitation, X-Ray Diffraction, SEM

Subject Areas: **Chemical Engineering & Technology, Nanometer Materials**

1. Introduction

Nanotechnology gained much attention for its vital pioneering role in manipulating materials at the atomic and molecular levels to dramatically alter the product properties. Materials reduced to the nanometric scale display

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Measurement of Forced Convective Heat Transfer Coefficient of Low Volume Fraction CuO-PVA Nanofluids under Laminar Flow Condition

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Abstract Experimental investigations of forced convective heat transfer coefficient of CuO-PVA nanofluids under uniform and constant heat flux are reported in this paper. Different nanofluid samples at different volume concentrations (0.05, 0.1 & 0.2%) were prepared by dispersing CuO NPs with an average size of 32.50 nm in 4 wt% PVA solution using ultrasonication and magnetic stirring. The forced convective heat transfer coefficient of the CuO-PVA nanofluids was measured with the help of vertical shell-and-tube heat exchanger where spiral circular copper tube was used. All the experiments were performed under laminar conditions ($Re \leq 2300$). The results under laminar flow conditions showed considerable enhancement of convective heat transfer with the use of nanofluids. There was increase in heat transfer coefficient of nanofluids CuO-PVA when compared with their base fluids. The increase is significant even though the concentration is less.

Keywords: *nanofluid, shell-and tube heat exchanger, forced convective heat transfer coefficient*

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1. Introduction

In recent times, nanotechnology has gained much attention for its vital pioneering role in manipulating materials at the atomic and molecular levels to dramatically alter the product properties. Materials reduced to the nanometric scale display significantly different properties compared to what they display at the macroscale or microscales. Because of their unique properties, nanomaterials are widely used in a variety of applications. Small amounts of nanoparticles can play a vital role in developing the properties of materials. Nanoparticles are becoming more and more important day by day as they play a beneficial role in a wide variety of scientific fields. In general, the size of a nanoparticle spans the range between 1 and 60 nm. Nanotechnology comprises the design, construction and utilization of functional structures with at least one characteristic dimension measured in nanometers. Currently nanoparticles are widely using in many fields [1-9].

Heat transfer enhancement is an active and important field of engineering research. Heat transfer fluids play an important role in many industries such as power generation, transportation, and newer electronic systems which have higher thermal requirements. Poor thermal conductivity is the main limitation in the development of energy efficient heat transfer fluids required for modern heat transfer equipment. The enhancement of heating or cooling in an industrial process may create a saving in energy, reduce process time, raise thermal rating and lengthen the working life of equipment. Most of the heat transfer fluids that are used in various applications have low thermal conductivity. Conventional fluids, such as refrigerants, water, engine oil, ethylene glycol, etc. have poor heat transfer performance and therefore high compactness and effectiveness of heat transfer systems are necessary to achieve the required heat transfer [10].

Generally metals, metal oxides and nanotubes are used in the preparation of nanofluids to enhance the thermal conductivity of the nanofluid by increasing conduction and convection coefficients [11,12]. Compared to other metal oxides oxides of copper have high thermal conductivity [13,14]. Copper oxide nanofluid shows reduced weight loss, enhanced thermal stability and thermal conductivity when compared to other fluids [15].

Thermal conductivity of commonly available and economically viable metal oxide nanoparticles is in the range of 10-40 W/m-K which is still two orders of magnitude higher than the thermal conductivity of water [16]. Thus, creating stable dispersions of nanoparticles in base fluids to improve thermal properties and thus heat transfer performance seems a logical approach.

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Characterization of Micro-fibrillated Cellulose Produced from Sawmill Wastage: Crystallinity and Thermal Properties

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Authors' contributions

This work was carried out in collaboration between all authors. Authors MSA and SYP designed the study, performed the instrumental analysis, wrote the protocol and wrote the first draft of the manuscript. Authors GMAK and MHU managed the analyses of the study. Author MAAM managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

In this work, micro-fibrillated cellulose (MFC) was prepared from sawdust and characterized by FTIR, WAXRD, and TGA. The MFC was synthesized by several steps such as alkali treatment, followed by $NaClO_2$ bleaching and acid hydrolysis. The acid hydrolysis was performed by three different H_2SO_4 concentrations (1N, 3N and 5N) whereas other conditions were remained unchanged. MFC were exhibited the identical peaks in FTIR spectra. The oxidation reaction took place during the MFC preparation by using high concentrations of acid which was detected by FTIR spectra. The crystallinity index of prepared MFC was measured by peaks at 16.2° and 22.2° (2θ

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ELECTROCHEMICAL CORROSION PERFORMANCE OF COMMERCIALLY USED ALUMINIUM ENGINE BLOCK AND PISTON IN 0.1M NaCl

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Abstract: *The corrosion behaviour of commercially used aluminium engine block and piston were investigated in 0.1M NaCl solution at room temperatures. The study was done by electrochemical method, using Tafel polarization and electrochemical impedance spectroscopy (EIS) techniques. The surface was characterized by optical microscope and scanning electron microscope (SEM). The results indicated differences in the charge transfer resistance of engine block and piston alloys. The current density (I_{corr}) of engine block material showed higher value than that of piston material. The corrosion potential (E_{corr}) and pitting corrosion potential (E_{pit}) of piston material were shifted to the more noble direction. In aluminium piston alloy there seems to be uniform surface pits formations which are in fewer amounts as compared to those in engine block alloy. The corrosion performance of aluminium piston alloy was found to be higher than that of aluminium engine block due to the presence of Ni and lower percentage of Fe in aluminium piston alloy.*

Keywords: *Localized corrosion, aluminium-silicon alloys, SEM, corrosion potential, polarization resistance.*

INTRODUCTION

Aluminum alloys with silicon as a major alloying element constitute a class of material, which provides the most significant part of all shaped castings manufactured, having a wide range of applications in the automotive and aerospace industries[1,2]. This is mainly due to the outstanding effect of silicon in the improvement of casting characteristics, combined with other physical properties such as mechanical properties and corrosion resistance. Recent articles on Al-Si alloys have reported that coarser dendritic structures yield higher corrosion resistance than finer dendritic structures, and that this is associated with the morphology of the interdendritic eutectic mixture. It was also reported that the silicon content is another important parameter affecting mechanical and corrosion resistances[3,4]. Corrosion is the gradual chemical or electrochemical attack on a metal by its surroundings such that the metal is converted into an oxide, salt or some other compound which results in loss of strength, hardness, toughness and other desirable mechanical properties. All corrosion reactions are electrochemical in nature and depend on the operation of electrochemical cells at the metal surface[5-7]. The corrosion resistance of aluminum is attributed to an exceptionally stable oxide film that forms on its surface. This film is resistant to attack from water and oxygen in a wide range of temperatures and pH levels, making aluminum alloys useful in a variety of environments. The adsorption of aggressive ions such as Cl^- into the faults in the protective film, and their penetration and accumulation in these imperfections is considered one of the triggering factors of the process of nucleation of pitting[8,9]. In addition, another factor which is associated with the susceptibility of aluminium to pitting corrosion and other forms of localized corrosion is the electrochemical nature of the intermetallic phases[10]. As a result, often the corrosion behavior can be correlated with the difference in potential between the matrix and the intermetallic compounds present in the alloy[11].

Corrosion studies of aluminium and aluminium alloys have received considerable attention by researchers because of their technological importance and industrial applications. Aluminium is second to iron in terms of production and consumption. Aluminium and aluminium alloys find applications, mainly in automobiles, aviation, household appliances; containers and electronic devices[12]. In the present paper, the processes of corrosion of a commercially available aluminium alloy engine block and piston in 0.1MNaCl solution were studied via electrochemical methods. The research has been directed towards studying the characteristics, condition of formation and morphology of attack.

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Investigation of the Structural, Dielectric and Electrical properties of Zn-substituted Li-Ni ferrite

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Abstract: *Substitution of Zn content on the structural, dielectric and electrical properties of $Li_{0.15}Ni_xZn_{0.35-x}Fe_{2-x}O_4$ (x = 0.00, 0.10, 0.20, 0.30 and 0.40) ferrites prepared by standard double sintering ceramic technique, sintered at 1150 °C for 5 hours have been studied. It has been observed that Zn-substitution on Li-Ni ferrites plays a remarkable role in improving its structural, electrical and dielectric properties. The bulk density, x-ray density and lattice constant of the samples decreases with increasing the Ni content. The porosity of the prepared samples didn't show any trend with Ni content. The X-ray diffraction (XRD) pattern confirmed the single-phase cubic spinel structure of the samples without having any other intermediate phases. The real part of permeability, Loss factor and AC resistivity have been found to be decreased while the Quality factor increased with the increase in Ni-content. Frequency dependent dielectric constant decreased with increasing the frequency as well as Ni-content.*

Keywords: *Standard double sintering ceramic technique, Lattice constant, Spinel ferrites, Permeability and Dielectric constant,*

I. Introduction

Ferrites constitute a special class of magnetic oxide materials with general formula $MO.Fe_2O_3$, where M is a divalent metal ion such as Mn^{2+}, Zn^{2+}, Ni^{2+}, Cu^{2+}, Mg^{2+}, Co^{2+}, Cd^{2+}. These materials show semiconducting characteristics which are of great technological applications since they have high electrical resistivity as well as better magnetic quantities [1]. Among the soft magnetic materials for high frequency power electronics Li-Ni ferrites is one of the most popular and smart candidate of highest rank [2]. These types of polycrystalline spinel ferrites have wide Varity of applications such as power electronics, ferro-fluid technology, magnetic data storage, in transformers, choke coils, noise filters and as contrast agent in magnetic resonance imaging [3-5]. It is well established that in Li-Ni ferrites most of the Ni^{2+} ions occupy in tetrahedral A sites [6] which results in a lowering of Fe^{3+} in octahedral A-sites. Moreover, Structural and other properties of ferrites are dependent on the quality of new materials, milling technique, preparation temperature, concentration of the dopants and impurities [7]. For these reasons, it is possible to introduce various metallic ions to these ferrites to improve their electronic and magnetic properties significantly [8]. In the last couple of decades a very large number of researches observed the effect of various additives on different properties of ferrites [9-32]. The effect of magnetic properties of Ni concentration in Li-Zn ferrite was studied by Soiban et al. [12]. From their investigation of saturation magnetization, Curie temperature and Mossbaur studies, it has been observed that Ni substitution greatly affects the magnetic properties of the Li-Zn ferrites system. Magnetic studies of Co substituted Li-Zn-ferrites were also studied by Soibam. Magnetisation measurements indicated that cobalt shows anomalous behavior when substituted in Li-ferrites in the presence of Zn [13]. The structure and magnetic properties of Mg-doped $Li_{0.43}Fe_{2.43}Mg_{0.17}O_4$ ferrites were investigated by Widatallah etal. [14]. The saturation magnetization and Curie temperature were found to decrease and the material increasingly turned super-parsmagnetic as milling proceeded at higher milling times. Tasakin et al. [15] studied the effect of sintering atmosphere without O_2 at high sintering temperature where high density was expected. This causes to decrease in permeability which might be attributed to the variation of chemical composition caused by volatilization of Zn. Rosses [17] reported that very high permeability is restricted to certain temperature ranges and the shapes of permeability versus temperature curves are strongly affected by any inhomogeneity in the ferrite structure.

Rezlescu [19] reported that the sintering behavior and microstructure of the ferrites largely affected by

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Extraction and characterization of alumina nanopowders from aluminum dross by acid dissolution process

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Abstract: A significant amount of aluminum dross is available as a waste in foundry industries in Bangladesh. In this study, alumina was extracted from aluminum dross collected from two foundry industries situated in Dhamrai and Manikganj, near the capital city, Dhaka. Aluminum dross samples were found to approximately contain 75wt% Al_2O_3 and 12wt% SiO_2. An acid dissolution process was used to recover the alumina value from the dross. The effects of various parameters, e.g., temperature, acid concentration, and leaching time, on the extraction of alumina were studied to optimize the dissolution process. First, $Al(OH)_3$ was produced in the form of a gel. Calcination of the $Al(OH)_3$ gel at 1000°C, 1200°C, and 1400°C for 2 h produced θ-Al_2O_3, (α+θ)-Al_2O_3, and α-alumina powder, respectively. Thermal characterization of the $Al(OH)_3$ gel was performed by thermogravimetric/differential thermal analysis (TG/DTA) and differential scanning calorimetry (DSC). The phases and crystallite size of the alumina were determined by X-ray diffraction analysis. The dimensions of the alumina were found to be on the nano level. The chemical compositions of the aluminum dross and alumina were determined by X-ray fluorescence (XRF) spectroscopy. The microstructure and morphology of the alumina were studied with scanning electron microscopy. The purity of the alumina extracted in this study was found to be 99.0%. Thus, it is expected that the obtained alumina powders can be potentially utilized as biomaterials.

Keywords: aluminum dross; alumina; nanoparticles; acid dissolution; calcination; X-ray fluorescence analysis

1. Introduction

Extraction of alumina has been of interest for several of its inherent properties, such as relatively high hardness, high abrasion resistance, and bioinertness [1–4]. Currently most of the world's commercial alumina is produced by the Bayer process using bauxite material [5]. Acidic and alkaline routes have also been reported for extraction of alumina. Acids such as H_2SO_4, H_2SO_3, and HNO_3 have been used as leaching agents [6–7]. In this study HCl was used as the leaching agent because of its several advantages; it is mild in its action, easily available, and produced as a byproduct in many chemical industries.

In Bangladesh bauxite is not available, but a significant amount of aluminum dross is obtained as a waste in its foundry industries. Aluminum dross is a complex oxide material containing Al_2O_3, SiO_2, Na_2O, Fe_2O_3, and other compounds. It is formed when molten aluminum comes in contact with air at the outer surface of the melt. It is a hazardous waste material [8]; however, it contains a high percentage of aluminum oxide, so it may be an alternative source of alumina. Recently an increasing number of alternative methods to the Bayer process have been developed in countries lacking bauxite deposits [9–10]. Namely, alkaline and acid processes have been proposed to produce alumina from aluminous ores where aluminum is dissolved from the ore as the aluminate ion (AlO_2^-). The Bayer process is quite

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Effect of Bismuth Addition on Structure and Mechanical Properties of Tin-9Zinc Soldering Alloy

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Abstract

Sn-Zn based solder is a possible replacement of Pb solder because of its better mechanical properties. The alloys need to be studied and explored to get a usable solder alloy having better properties. In this work eutectic Sn-9Zn and three Tin-Zinc-Bismuth ternary alloys were prepared and investigated their microhardness and mechanical properties. Microhardness, tensile strength and elastic modulus increase with Bi addition while ductility decreases with Bi addition.

Keywords

Lead Free Solder Alloy, Eutectic Alloy, Microhardness

1. Introduction

Soldering alloy is the prime material for electronic packaging. Selecting proper material for soldering is important. Melting temperature, mechanical properties, wetting properties and low cost, etc. are very important issues for selecting a solder material. Tin-Lead solders have been widely used in electronic and optoelectronic packaging due to their low cost and low melting temperature and good soldering properties. But lead is an aggressive threat for human health and the environment due to its toxicity. Many countries banned using lead and lead alloys [1] [2] for their use in packaging. Waste Electronic and Electronic Equipment (WEEE) and Restriction of Hazardous Substances (ROHS) approved banning the use of lead in EU countries from July 2006. The USA, the EU and Japan forbade the use of Lead containing products [3] [4]. Au-Sn is thought as an alternative but its

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Thermal and Electrical Properties of Sn-Zn-Bi Ternary Soldering Alloys

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Abstract

Sn-Zn based solder is a possible replacement of Pb solder because of its better mechanical properties. The alloys need to be studied and explored to get a usable solder alloy having better properties. In this work, eutectic Sn-9Zn and three Tin-Zinc-Bismuth ternary alloys were prepared and investigated their thermal and electrical properties. Thermo-mechanical Analysis and Differential Thermal Analysis were used to investigate thermal properties. Microstructural study is carried out with Scanning Electron Microscope. The alloys have single melting point. The co-efficient of thermal expansion and co-efficient of thermal contraction varies with alloy composition and temperature range. Electrical conductivity changes with Bi addition.

Keywords

Lead Free Solder Alloy, Eutectic Alloy, DTA, TMA, Conductivity

1. Introduction

Tin-Lead solders have been widely used in electronic and optoelectronic packaging due to their low cost and low melting temperature and good soldering properties. But lead is an aggressive threat for human health and the environment due to its toxicity. Many countries banned using lead and lead alloys [1] [2] for their use in packaging. Waste Electronic and Electronic Equipment (WEEE) and Restriction of Hazardous Substances (RoHS) approved banning the use of lead in European Union countries effective July 2006. The USA, the EU and Japan forbade the use of Lead containing products [3] [4]. Because of the toxicity of lead, traditional Sn-Pb solders are now being replaced with Sn-base soldering alloys containing additions of other metals (Ag, Bi, In, etc.) [5]-[9]. Au-Sn is thought to be alternative but its mechanical properties are not sufficient [10] [11]. SAC

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Development of functionalized carbon nanotube reinforced hydroxyapatite magnetic nanocomposites

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Magnetic properties

ABSTRACT

An innovative and effective approach is introduced to functionalize multi-walled carbon nanotubes (f-MWCNTs) by in-situ chemical precipitation of hydroxyapatite (HA) to improve their magnetic properties. The HA/f-MWCNTs nanocomposites are obtained by pressureless sintering in vacuum atmosphere. The carboxyl functional group (–COOH) is introduced by an acid treatment on the MWCNT surface. Magnetic hysteresis measurement reveals that HA/f-MWCNTs nanocomposites exhibit excellent hard ferromagnetic properties with saturation magnetization (M_S) of 0.233 emu/g and coercivity (H_C) of 2985.53 Oe at room temperature. The maximum magnetic hysteresis loss of 0.44 kJ/m^3 induces an expected heat generation and it is expected that this nanocomposite has potential to be used as a biomaterial for hyperthermia treatment of bone cancer and other biomedical applications.

1. Introduction

In recent years, assembling nanomaterials into special nanostructures has gained considerable scientific attention because of their extensive range of biomedical applications, such as magnetic resonance imaging, drug-delivery, as well as the thermo-seeds embedded in bioactive materials which are utilized for bone cancer treatment using the hyperthermia method [1–3]. Magnetic nanomaterials have frequently been used for the purpose of hyperthermia treatment and the killing of cancer cells in bones, which involves localized heating of infected parts of the body by applying an alternating magnetic field. But magnetic nanoparticles are prone to aggregation and rapid biodegradation when they are exposed to a biological system [4] which can be problematic considering the importance of biosafety of the nanoparticles in medical applications. The materials that are compatible with bone tissue are preferred for the treatment of bone repair and related cancer therapy. Hydroxyapatite (HA; $Ca_{10}(PO_4)_6(OH)_2$) is a biologically active material that has drawn great attention from researchers due to its excellent biocompatibility, osteoconductive properties and a chemical structure similar to apatite in the human skeletal system [5–7]. Hydroxyapatite is widely applied as a biomedical material, including such uses as bone fillers, hard and soft tissue repairs, drug and gene delivery systems, protein separation and column chromatography for rapid fractionation of biomolecules [8–12]. In addition, HA materials are also potential candidates for use in cell targeting, near-infrared (NIR) fluorescence labeling, imaging and diagnostic materials [13,14]. Recently, there is growing interest in hydroxyapatite-based nanocomposites with a magnetic property as drug delivery carriers to precisely target the desired organs or tissues inside the body and thus significantly reduce unnecessary damage to healthy tissue by applying local heat through an external magnetic field [15]. Carbon nanotubes (CNTs), especially multi-walled carbon nanotubes (MWCNTs) have been considered as potential candidates for a reinforcing agent of hydroxyapatite due to their unique thermal, electronic, magnetic properties and as innovative carriers for bone morphogenetic protein [16–18]. Moreover, CNT-based biomaterial composites are proven to be suitable for cell growth and growing enzyme activity [19,20]. Functionalized carbon nanotubes (f-CNTs) act as carriers for the delivery of a wide range of therapeutic agents [21]. The healing potential of a defective bone is significantly enhanced when therapeutic agents are delivered through HA nanocarriers [22,23]. Therefore, it is important to develop the HA/f-MWCNTs nanocomposites with an improved magnetic property so that they can be used as a thermal seeds by magnetically induced hyperthermia, killing cancer cells at temperatures >43 °C for bone cancer treatment. To maximize the effects of MWCNTs for the aforementioned biomedical applications, they have to be functionalized or chemically modified in order to improve their biocompatibility by the presence of

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Epoxide Functional Temperature-Sensitive Semi-IPN Hydrogel Microspheres for Isolating Inorganic Nanoparticles

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ABSTRACT: In this investigation, epoxide (oxirane), a cyclic ether with a three-atom ring, functional semi-interpenetrating polymer network (semi-IPN) hydrogel microspheres composed of temperature-sensitive poly(*N*-isopropylacrylamide-*N,N*-methylene-bis-acrylamide) (P(NIPAM-MBA Am)) and P(NIPAM-glycidyl methacrylate) (P(NIPAM-GMA)) are prepared. We called these microspheres as P(NIPAM-MBA Am)/P(NIPAM-GMA). The microspheres exhibited temperature-sensitive volume phase transition at a temperature range of 33–35°C. The chemical bonding of anticancer drugs with the epoxide ring increased the absorption capacity but their release is affected, as the temperature is decreased below the volume phase transition, studied under an in vitro condition suitable for application in the treatment of cancer cells. The epoxide functionality of semi-IPN hydrogel microspheres is also utilized to isolate inorganic nanoparticles, using Fe_3O_4 nanoparticles as an example, simply by blending the hydrogel and magnetic (Fe_3O_4) dispersions. The presence of strained oxirane ring derived from GMA segment induced high affinity for semi-IPN hydrogel microspheres toward magnetic Fe_3O_4 nanoparticles and eventually reduced the colloidal stability. © 2015 Wiley Periodicals, Inc. Adv Polym Technol 2015, 0, 21645; View this article online at wileyonlinelibrary.com. DOI 10.1002/adv.21645

KEY WORDS: Anticancer drug, Epoxide, Nanoparticles, Semi-IPN, Temperature-responsive

Introduction

Stimuli-responsive hydrogels exhibiting sharp volume phase transition in response to small variation in environment stimuli such as temperature, pH, ionic strength, electric current, redox, light, etc. are attractive soft materials.[1-17] These materials are drawing much attention from the researchers for their wide range of biomedical and pharmaceutical applications.[18-24] Temperature is one of the important triggering signals for volume phase transition among the reported stimuli-responsive hydrogels. Cross-linked poly(*N*-isopropylacrylamide) (PNIPAM) is a widely studied temperature-responsive hydrogels. These PNIPAM hydrogels reportedly show a volume phase transition temperature commonly known as lower critical solution temperature (LCST) at about 34°C.[25,26] At temperature below the LCST, the PNIPAM hydrogels swell due to the formation of hydrogen bonds among hydrophilic amide groups and surrounding water molecules. While as the temperature increases above the LCST, the PNIPAM hydrogels dehydrate because the hydrogen bonding interactions either weaken or disrupt and the hydrophobic interactions among the hydrophobic isopropyl groups dominate.

The poor mechanical strength of these cross-linked PNIPAM hydrogels particularly in the swollen state often reduces their application potentials outside the biomedical field. The strength of hydrogels can be increased by incorporating cross-linking agents, comonomers, and increasing the degree of cross-linking. Recently numerous semi-interpenetrating polymer network (semi-IPN) hydrogels with improved mechanical properties have been reported.[27-35] Semi-IPN hydrogels are defined as a mixture of two or more interwinding polymers where one of the network polymers is linear and is synthesized in

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Use of Magnesium Sulphate and Boric Acid to Reduce Ecologically Unfavoured Ammonium Sulphate as Deliming Agent in Leather Processing

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Abstract

The use of an alternative deliming agent in leather processing is described which will reduce or in some cases completely eliminate the use of ammonium salts, thereby reducing the level of nitrogenous compoents in the tannery effluent. To choose the suitable deliming agent magnesium sulphate and boric acid are used separately or/and their mixture with ammonium sulphate in the deliming step of leather processing. After analysing physical and chemical properties of the leather it is found that boric acid delimed hides are best quality and in this process uptake of chrome is also better.

Introduction

When the hides and skins are unhaired and fleshed they are in a swollen and pumped condition and are full of lime. The limed hides and skins are called pelt. Before the pelt can be satisfactorily tanned to produce leather it is necessary to free them from the lime. Lime content in the pelt may vary (from 0.5-2.0 %) on pelt weight.[1]

Ammonium sulphate is commonly used to delime hides and skins in tanning industries. A practice that results in elevated levels of ammonium in beam house effluent and create unpleasant working conditions. But the maximum permissible limit of ammonia nitrogen in the industrial effluent is 50 mg/L.[2] So, the disposal of ammonia containing effluents into nearby water resources poses an eminent danger especially in regard to maintenance of fish life. Excessive levels of ammoniacal nitrogen also cause water quality problems.[3] Besides, these dissolved ammonia escapes to the atmosphere when the effluents are discharged, which cause adverse health effects on humam life like respiratory irritations, severe pulmonary irritations and skin irritations.[4]

From the literature survey it is found that some works have already been done by other scientists to replace ammonium sulphate as a deliming agent using magnesium lactate and magnesium chloride in combination with sulphuric acid or hydrochloric acid. But their introduction into the float, however, can create localized areas of low pH. This can cause acid shock and result in drawn grain

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Fatliquor preparation from Karanja seed oil (Pongamia pinnata L.) and its application for leather processing

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Abstract: Fatliquor affects the physical properties of the leather and makes more flexible and softer. Fatliquor prepared from karanja seed oil by sulphation process followed by addition of sodium hydroxide to maintain pH at 5.0 with conc. sulphuric acid. FT-IR analysis of the sulphated product confirmed the attachment of sulphonic acid group. The physical and chemical properties of the fatliquor were found satisfactory. The prepared fatliquor was applied for the processing of goat skins. The processed goat skins physical and chemical properties compared with the skins processed by fatliquor made from castor oil and all the remaining leather processing chemicals same in both processing. Goat skins processed by these two types of fatliquor found standard leather specification. So fatliqour prepared from karanja seed oil extracted by soxlet extraction method considered as a substitute imported fatliqour.

Keywords: Fatliquor, FT-IR, Strength properties, organo leptic properties, sulphation, Karanja seed oil.

I. Introduction

Karanja (Pongamia pinnata L.) belongs to the family of Leguminaceae. These trees are normally found around coastal areas, river banks, tidal forests and road sides. Karanja is one of the nitrogen fixing tree produces seed kernels containing 27-39 percent of oil. The fresh extracted oil is yellowish orange to brown colour and rapidly darkens on storage. It has a disgusting odour and bitter taste. Karanja oil is mainly used as a raw material for soap manufacturing but the main constrains for its other usage are the color and odor [1]. The oil is used mainly as lubricant, water-paint binder and production of biodiesel by methanolysis [2].

This paper describes the use of Karanja seed oil for the preparation of fatliquor and its uses for leather processing. Most of the fatliquors are imported from other countries and has invested huge foreign currency on imports. In this study we try to prepare substitutes of imported fatliquors by the fatliquors prepared from our local raw materials, Karanja seed oil.

The fatty acid composition of typical Karanja seed oil is shown in the Table-1.

Table-1: The fatty acid composition of a typical karanja seed oil [2]

Fatty acid	Percentage
Palmitic acid (C_{16})	11.65
Stearic acid (C_{18})	7.50
Oleic acid ($C_{18:1}$)	51.59
Linoleic acid ($C_{18:2}$)	16.64
Eicosanoic acid (C_{20})	1.35
Dosocasnoic acid (C_{22})	4.45
Tetracosanoic acid (C_{24})	1.09

From the table it is shown that in Karanja seed oil contains higher proportion of oleic acid than other fatty acid. Higher proportion oleic acid content is suitable for fatliquor preparation

II. Material And Methods

2.1 Raw material

Karanja seeds were collected from Rajshahi district, Bangladesh and full grain chrome tanned leathers were collected from commercial hide brokers.

2.2 Reagents

Reagents used for analysis are laboratory or analytical grade and leather processing are commercial grade.

2.3 Oil extraction

Karanja seeds are grained to powder form and shade dried for 10-15 days. The dried powders are subjected to Soxlet extraction using n-hexane as a solvent.

2.4 Sulphation process

A mixture of 40 ml concentrated sulphuric acid and 10 ml of concentrated phosphoric acid are added drop wise in 200 gm of karanja oil with constant stirring at 18-20°C temperature. Reaction was carried out slowly (approximately 3 hours required to complete the reaction). The sulphated products shaken with 400 ml of 10% sodium chloride solution and then keep in a separating funnel overnight and layer was separated. The pH of the separated upper layer sulphated liquor was neutralized at pH 5.0 by adding 30% sodium hydroxide solution and the resulting fatliquor then applied for leather processing.

2.5 Analysis of the fatliquor

Karanja seed oil was analysed and its chemical and physical characteristics are characteristies according to the American Oil Chemists Society Methods [3]. Refractive index, acid value, saponification value and unsaponificable matter were measured.

2.6 FT-IR analysis of Sulphated Product

In order to investigate presence of sulphonic acid group in sulphated fatliquor FT-IR spectra of sulphated fatliquor was taken using a Shimadzu FT-IR [4].

2.7 Fatliquoring process

After shaving the leather at 0.9 mm, it was wet back by adding water and wetting agent in the drum. After 30 minutes run the water was drained off. Then by adding 2% neutralizing syntan in the drum it was run for another 15 minutes at approximately 10 rpm in the ambient temperature. There after 0.5% sodium bicarbonate was added and the drum was run for an additional 15 minutes at the same speeds maintaining the pH at 5.0-5.5. The neutralized leather was washed with water and dyed with 1% acid dye for 30 minutes [5], [6].

Then 6% fatliquor was added to the dye bath at room temperature and was run at 10 rpm for 40 minutes. The dyed leather was then washed with water and removed from the drum. After samming and settling out it was air dried at room temperature and was used for various physical properties investigation. The fat liquoring process of skin is illustrated by the Table-2.

Table-2: Practical recipe for fat liquoring of skin

Process	%	Product	Duration (min)	Remarks
Wet back	200	Water at 35°C		
	0.2	Ethoxylated Fatty Alcohol		
	0.2	Oxalic Acid	Drum 30 min	pH 3.8
Neutralization	100	Water at 35°C		
	0.5	Sodium bicarbonate		
	2	Neutralizing Agent	Drum 30 min	pH 4.6
Dyeing-Fatliquoring	60	Water at 30°C		
	1	Dye (Beige)	Drum 30 min	
	100	Water at 60°C		
	6	Sulphated Vegetable Oil (Karanja oil)	Drum 60 min	
	1.0	Formic Acid	Drum 10 min	
	1.0	Formic Acid	Drum 10 min	pH 3.9
			Run-off and Wash 10 min	

III. Result and Discussion

3.1 Analysis of Karanja seed oil

3.1.1 Physical properties

The physical properties of Karanja seed oil, sulphated Karanja seed oil and sulphated Castor oil (which is conventionally used as fatliquor in leather processing) are shown in Table-3.

Table-3: Physical properties of Karanja seed oil, sulphated Karanja seed oil and sulphatedn Castor oil

Parameter	Karanja oil	Sulphated Karanja oil	Sulphated castor oil
Appearance	Dark brown liquid	Brown liquid	Pale amber liquid
Solubility in water	Insoluble	Soluble	Soluble
Refractive Index (20.5° C)	1.4686	1.467	1.479
Specific Gravity at (20.5°C)	0.880	0.861	1.029

Mean ± SD triplicate analysis

From the table it can be shown that the appearance of sulphated Karanja seed oil and sulphated Castor oil are distinct. Both the sulphated oil is soluble in warm water. The refractive index value, specific gravity values are higher in case of sulphated castor oil. Thus the physical properties of Karanja seed oil are in agreement with the reported values [3], [7].

3.1.2 Chemical properties

The chemical properties of Karanja seed oil and sulphated Karanja seed oil compared with sulphated Castor oil are shown in the Table-4.

Table-4: Chemical properties of Karanja seed oil, sulphated Karanja seed oil and sulphated Castor oil

Parameter	Karanja oil	Sulphated Koranja oil	Sulphated castor oil
pH of 10% solution	8.1	6.2	7.0
% Fatty matter	91.04	60.9	70.1
Saponification value	185	187	176
Iodine Value	86.5	81.5	81
Acid Value	7.1	5.06	8.0
SO_3 content	–	4.1%	5.1%
Ash content	–	6.2%	2.8%

Mean ± SD triplicate analysis

From the table-4 it is shown that Karanja seed oil contains high percentage of fatty matter, which indicates the use of this oil for preparation of fatliquor.

Iodine value shows that the oil has high quantity of unsaturated fatty acid. The iodine value is used to determine the degree of unsaturation of fatty acids. It is also reported that the oleic acid is the main fatty acids of this oil.

Karanja seed oil has been found to have the higher saponification value (Table-4). Since saponification value is inversely proportional to the molecular weight of the fatty acid present in oil. So it is found in liquid form at room temperature. Karanja seed oil also represents the higher acid value which makes this oil suitable ingredient for fatliquor.

Colloidal emulsion is indicative of low to medium degree of sulphation. This physical observation confirms that there is percentage of SO_3 incorporated in the sulphated compound. The combined SO_3 or emulsifier is the fuel which drives the oil droplets into the leather. Anionic emulsifier ensures a great degree of fixation [8], [9] since they will be attracted to the positively charged leather.

It is found that the iodine value of the raw Karanja seed oil decreases after sulphation, while the acid value increases showing the hydrolysis of fatty molecule.

The pH value of sulphated Karanja seed oil and sulphated Castor oil was found to be 6.2 and 7.0 respectively. This may result of neutralization carried out during the sulfating process.

The result showed that in sulphated Karanja seed oil the percentage of organically combined SO_3 [6] is relatively lower than the sulphated castor oil.

The sulphated Karanja seed oil showed higher ash content than sulphated Castor oil. This value referred to the sodium salt produced by neutralization of acid during sulphation.

3.2 FT-IR Analysis [4]

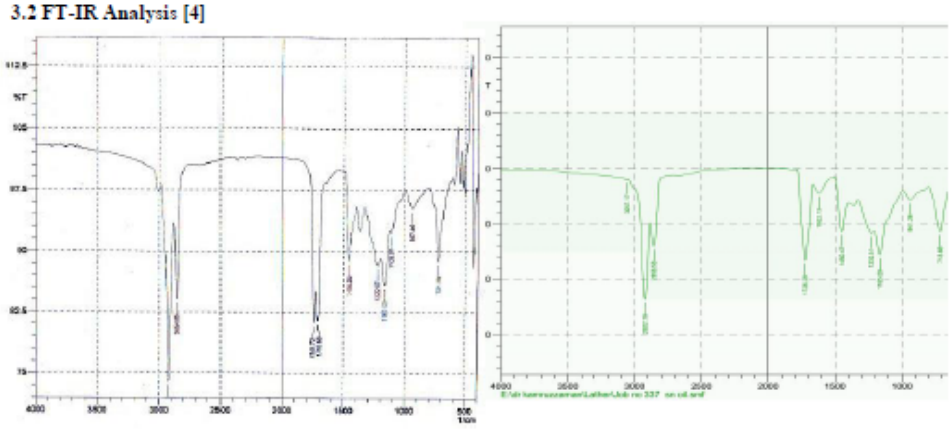

Fig. 1(a) The FT-IR spectra of sulphonated oil **Fig. 1(b).** FT-IR spectra of unsulphonated oil

Fig. 1(a) & 1(b) illustrates the IR spectrum of sulphated and unsulphated Karanja seed oil (Pongamia pinnata L.). The peaks at Fig.1(a)2854.65cm^{-1}, 1165.00cm^{-1} & 1222.87cm^{-1} represent the presence of C-H, C-O & -SO_3 groups respectively.

The peaks at Fig.1(b). 2922.16cm^{-1}, 1726.29cm^{-1} & 1165.00cm^{-1} are due to the presence of =C-H, C=O & C-O respectively.

3.3 Stability of Karanja seed oil

The stability of the prepared fatliquor in different solution is shown in Table-5.

Table-5: Stability of 10% solution

Solution	Sulphated Karanja seed oil
5% NaCl	Stable
5% Na_2SO_4	Stable
5% Basic Chromium Sulphate	Stable 1-2 hours
5% MgO	Stable
5% Formic Acid	Not stable

From the Table it can be shown that the prepared fatliquor is stable in salts, tanning and basification agent. This makes it possible using in re-tanning and fatliquoring steps. The stability of the emulsion of the experimental fatliquor is due to the poly hydroxyl sulphate group which is resistance to hydrolysis.

Commercial sulphated oil may give either a solution or emulsion with water. Such an emulsion may be either transparent or opaque. This variation in types of emulsion is dependent on the degree of sulphation and to a subsequent treatment and the neutralization. In general the more opaque types of conclusion are considered to due taken up better in fat-liquoring. The prepared fat-liquor gives colloidal emulsion with warm water.

3.4 Strength properties

The strength properties [10] like tensile strength tongue tear strength were tested using an instron tensile tester and grain crack & grain brust using lastometer of two matched side leather those were fat liquored by using sulphated Karanja seed oil and imported sulphated castor oil have been compared which is shown in the

Table-6: Strength Properties

Properties		Prepared fat-liquor	Imported fat-liquor
Tensile strength (N/mm^2)	Parallel	15.05	19.03
	Perpendicular	17.93	21.40
Elongation at break (%)	Parallel	53.0	49.0
	Perpendicular	28	24
Tear resistance (nm)	Parallel	4.9	5.01
	Perpendicular	7.2	7.11

From the strength properties analysis conducted, it is evident that the leather fatliquored using sulphated Karanja seed oil comparable to sulphated castor oil.

Some difference was observed in the physical test of leather however, results were found under the limit of standard specification which confirmed [6], [8] the suitability of prepared fatliquor. As the strength properties of the leather are also decided by the Cr_2O_3 and fat content, the leather have been physically tested for tensile strength, elongation at break, stitch tear strength and the results are reported in table-6.

The chromic oxide content and fat content in leather seems to have acceptable value for both the sulphated Karanja seed oil and Castrol oil. The enhancement in mechanical properties of treated leather using sulphated Karanja seed oil is due to good lubrication of [6], [11] fibers. The prepared fat-liquor possesses good penetration power and emulsion stability.

Tensile strength is the value of load the sample cross section can bear when load by the axial load and it is related with the leather sample state of collagen fibers. Therefore, the tensile strength can characterize the flexibility of fibers beside for the strength of collagen fibers.

Flexibility is one of the most important properties of leather achieved from fat-liquor. Fat-liquor reduce the friction between fiber is mainly related to the nature of sulfated oil and the quality of introduced oil. The elongation at break can characterize the softness, flexibility [5], [6], strength and toughness of leather matrix.

3.5 Physical testing and Hand Evaluation of leather

Experimental and control crust leather were assed for fullness, softness, grain tightness (break) by hand and visual examination. The leathers were rated on a scale of 0-10 points for each functional property by experienced leather technologist, where higher points indicate leather property.

3.6 Organo leptic properties

Leather made with sulphated Karanja seed oil and imported fatliquor based on castor oil were analysed for their organo lepltic properties which are shown in the Table-7.

Table-7: Organo leptic properties

Properties	Trial with prepared fatliquor	Trial with imported fatliquor
Softness	3.5	4.5
Fullness	4.0	4.5
Surface touch	4.5	4.5
Grain tightness	4.5	4.5
Odor	Odorless	Distinct

From the Table it can be shown that the leather made with the sulphated Karanja seed oil has the similar softness, fullness with the fatliquor based on Castor oil. Leather will be full if fiber sickening of the tanned fiber structure is well avoided.

Firmness or looseness of the grain seems also be coupled with the amount of fat [12], [13] in the grain. If the papillary layer is externally softened the grain may become loose and poor break occurs. For fullness, firmness is more critical issue; it requires proper adjustment of combination of fatliquor, dyeing and subsequent mechanical operation.

IV. Conclusion

The leather processed by the prepared fatliquor and conventional fatliquor are very closer in respect of physical, chemical, strength and organo leptic properties. Firmness or looseness of the grain coupled with the amount of fat in the grain. If the papillary layer is externally softened the grain may become loose and poor break occurs. For fullness, firmness is more critical issue; it requires proper adjustment of combination of fatliquor, dyeing and subsequent mechanical operation. Firmness value of prepared fatliquor less than convention liquor firmness value but higher percentage of fatty matter content ensures its suitability for preparing fatliquor. On the other hand prepared fatliquor is stable in salt, tanning and basification agent. Moreover the physical and chemical properties of the prepared fatliquor are very much closer to those of conventional fatliquor. Thus Karanja seed oil may be considered as an important source of fatliquor material.

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Studies on the Tanning with Glutaraldehyde as an Alternative to Traditional Chrome Tanning System for the Production of Chrome Free Leather.

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Abstract

Chrome tanning is a versatile tanning system, but the pollution problems of chromium due to inadequacy of treatment systems and possible formation of Cr(VI), a carcinogen, have led to search for an eco-friendly and viable alternative tanning system. The present investigation focuses on the development of a novel, high performance and thermally stable aldehyde tannage system to produce chrome free leather by cross linking the NH_2 groups of collagen with glutaraldehyde ($CHO\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}CHO$). After as usual soaking, liming, deliming, bating and pickling operations the pickle pelts were tanned with glutaraldehyde and other syntans. This developed chrome free tanning process produce crust leathers exhibiting thermal stability >85^{O}C and reducing the TDS value with 0% emission of Cr-salt in tannery discharge.

Key words : Eco friendly tanning system, Chrome free leather, Crust leathers

Introduction

The tanning industry is one of the oldest and fastest growing industries in South and Southeast Asia but tightening environmental regulations have greatly impacted the operation of tanneries throughout the world. This is because in the leather industry from 1000 kg of raw hides, yield only 150 kg leather and the remaining are 150 kg split, 700 kg solid waste and 30 m^3 of effluent containing 400 kg dissolved and suspended wastes. In this huge amount of tannery waste, chromium salts are considered to be the most heinous element which is used in chrome tanning stage as basic chromium sulphate. So polluting nature of chromium salts has placed chrome tanning under severe criticism owing to ecotoxicological objections, although chrome tanned leathers do possess

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many significant advantages. For these reasons a viable alternative to chrome tanning system is of paramount interest for the production of leathers similar to those of conventional chrome tanned leathers. From the updated scientific literature survey it is found that many attempts have been taken to produce chrome free leather using iron-complexes, synthetic resins with aluminium and titanium, tara-aluminium tanning, wet white pretannage and aldehyde/polymer combination etc. (Balasubramanian and Gayathri, 1997; Gangopadhyay *et al,* 2000; Palop, 2003; Vitolo *et al,* 2003; Palop, 2000). But these processes suffer from various disadvantages such as aluminium tannnage gives inferior shrinkage, and light-fastness, titanium tannage gives full leathers with yellowish tinge, titanium tannins are also expensive. Using vegetable tannins the leather can not achieve the characteristics like chrome tanned leather.

The research reported in this paper seeks to overcome the above disadvantages using glutaraldehyde as a pretanning agent and to minimize the environmental impact of chrome tanning.

Materials and Methods

Raw materials

The brined goat skins and cow hides were obtained from commercial hide brokers. Several experiments were carried out using freshly flayed goat skins of average weight 1kg and average areas 5 sq. ft. and cow hides of average weight 10 kg and average area of 20 sq. ft. obtained from local slaughter houses.

Equipments

In the pretanning step stainless steel drum of 18" width and 36" diameter were used. The speed of the drum was 4 to 20 rpm. For subsequent operations upto crust and finish as usual machinaries were used.

Chemicals

The following chemicals were employed in the pretanning step of leather processing glutaraldehyde, sodium meta bi-sulphite, syntan, sodium bi-carbonate and fungicide.

All the chemicals were commercial grade products. The chemicals employed in subsequent operations were those normally used in leather industry.

Optimization of glutaraldehyde for pretanning

The percentage glutaraldehyde offer was varied from 0.5% to 3% for tanning along with the fixed amount of the syntan and other chemicals. Pickled pelts were used for tanning experiments. The shrinkage temperature of various tanned leathers were determined and benchmarked for selecting the optimum amount of glutaraldehyde.

Tanning processes

For both glutaraldehyde tanning and chrome tanning operations of leather processing the

raw hides were converted into pickled pelt by traditional beam house operations. Then the pickle pelts were converted into wet white and wet blue leather and subsequently to crust leather by the following processes.

Table-I. Processing steps

Glutaraldehyde tanning for the production of crust leather	Chrome tanning for the production of crust leather
Tanning by 2.5% glutaraldehyde, 1 % sodium meta bi- sulphite, 50% water, 2% naphthalene based syntan, 0.6% sodium bi-carbonate and 0.2% fungicide.	Tanning by 6% basic chromium sulphate and 2% chrome syntan.
Then samming, splitting and shaving operations were done for the production of wet white leather.	Then samming, splitting and shaving operations were done for the production of wet blue leather.
Neutralization by 1.5% Neutralizing syntan. Retanning by 4% acrylic syntan, 6% vegetable tannin and 4% resin syntan.	Neutralization by 1.5% Neutralizing syntan. Retanning by 4% acrylic syntan, 6% vegetable tannin and 4% resin syntan
Then fatliquoring and dyeing were done for the production of crust leather.	Then fatliquoring and dyeing were done for the production of crust leather.

Determination of shrinkage temperature

Samples of tanned leather were tested in the wet state, just after tanning. This means that immediately after tanning the specimens were washed in running water and squeezed several times with a blade to eliminate the excess water and tanning solutions. Triplicate leather samples for a total samples dimension of 50 x 3 mm for a thickness less than 0.3 mm were used for shrinkage temperature determination. The method used for shrinkage temperature determination is codified in the international norm (International leather union) for leather. The heating rate used was of 1.50 C/min.

Tanning discharge analysis

The waste water discharge from tanning operation was analyzed for some common characteristics of waste water which are important in assessing the potential for water pollution like pH, BOD, COD, total solids, dissolved solids, suspended solids, chloride, total chromium etc. according to the standard method (Clesceri *et al,* 1999).

Sampling for physical and chemical analysis

In each experiment the hides and skins were sampled after each processing stage (Standard practice for sampling, 2002. For physical testing square (10" x 10") samples were taken 4" away from the foot of the tail and 2" away from the back bone. For chemical analysis rectangular pieces of leather measuring 6.5" x 2.5" were taken from each

side representing the butt, belly and shoulder portions.

Physical analysis

The main physical properties namely thickness of the leather, tensile strength, extension, ball brust of controlled leather and those of tanned with glutaraldehyde, conditioned at 80 ± 4° F and 65 ± 2 % R. H. for 48 hours are assessed in accordance with standard methods (Dutta, 1999) Each value reported is an average value of four samples.

leather To optimize the amount of glutaraldehyde for tanning different amounts of glutaraldehyde were used and the results of shrinkage temperature and visual assessment data of leather are shown in Table II.

From the table it is evident that with increase in the amount of glutaraldehyde added considerable increase in shrinkage temperature of leather was obtained using glutaraldehyde upto 2%, with further increase in glutaraldehyde to 2.5% there is only slightly increase in shrinkage temperature of tanned leather.

Table-II. Shrinkage temperature and visual characteristics of leather at different percentages of glutaraldehyde

Glutaraldehyde added (%)	Shrinkage temperature (Ts°C)	Visual assessment scale (0-10)*	
		Grain characteristics	Fullness
0.5	80	8	5
1.0	80	7	6
1.5	82	7	7
2.0	84	7	8
2.5	85	7	9
3.0	85	6	9

* The scale of 0-10 indicate 0 for poor and 10 for very good.

Chemical analysis

Volatile matters, ash, hide substance, chrome content of both the leathers tanned with glutaraldehyde and chrome were determined by the official methods of analysis of SLTC (Society of Leather Technologist and Chemists, 1996).

Results and Discussion

Effect of glutaraldehyde on the shrinkage temperature and visual characteristics of

Further increase in glutaraldehyde offer results no change of shrinkage temperature.

The increase in the offer of glutaraldehyde to 3% results in the leather of coarse gain. Empty leathers have been obtained when 0.5% glutaraldehyde was used and the fullness of the leather increases with the increase of glutaraldehyde added upto 2.5%. Considering both the shrinkage temperature and the properties of the tanned leather, 2.5%

glutaraldehyde (based on the pelt weight) has been found to be the optimum offer.

Tanning discharge analysis

The discharge from chrome tanning and glutaraldehyde tanning were analyzed for the parameters which are listed in Table III.

From the table it is found that though the BOD and COD in the discharge of glu taraldehyde tanning is high but the total solids and chloride are much less in glutaraldehyde tanning discharge. From the table it is revealed that there is no discharge of chromium in the effluent of glutaraldehyde tanning. So in environmental aspects the glutaraldehyde tanning is much more acceptable than the conventional chrome tanning.

Table-III. Characteristics of tanning discharge.

Parameter	Chrome tanning	Glutaraldehyde tanning
pH	2.5 - 4.0	3.0 - 4.0
BOD 5 day at 20^{O}C (Total)	350 - 800	500 - 1000
COD (Total)	1000 - 2500	1200 - 3000
Total solids (TS)	30,000 - 60,000	20,000 - 40,000
Dissolved solids (DS)	29,000 - 57,500	25,000 - 48,500
Suspended solids (SS)	1000 - 2500	800 - 1800
Chloride (as Cl)	15000 - 25000	13,500 - 22,000
Total chromium (as Cr)	1500 - 4000	0

All the values except pH are expressed in mg/L.

Physical parameters

The physical parameters of both glutaraldehyde tanned and chrome tanned leathers are listed in Table IV. From the table it is found that the physical properties of the glutaraldehyde tanned leather is comparable or in some cases better than the leather tanned by conventional chrome.

Chemical properties

The chemical properties of the leather tanned by glutaraldehyde and chrome are shown in Table V. From the table it is found that in wet white leather percentage of Cr_2O_3

Table : IV. Physical properties of the tanned leather

Physical properties	Glutaraldehyde tanned leather	Chrome tanned leather
Thickness (inches)	0.065	0.064
Tensile strength (psi)	2256.3	2255.80
Extension (%)	44.10	45.21
Ball brust (lbs)	124.50	25.10

Table :V. Chemical properties of the glutaraldehyde tanned leather and chrome tanned leather after shaving

Assessment	Chemical composition of pretanned leather after shaving (wet white)	Chemical composition of chrome-tanned leather after shaving (wet blue)
Volatile matters	9.8%	3.0%
Sulphated total/ash	1.3%	2.3%
Hide substance	29.5%	75.2%
Cr_2O_3	---	4.8%

is nill because no chrome is used in wet white leather. In wet white leather ash and hide substance is less than chrome tanned leather but percentage of volatile matter is higher in wet white leather.

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Development of hair-save unhairing method using organic thio compounds in pre-tanning stages of leather production.

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Abstract

The function of liming and unhairing is to remove hair, interfibrillary components, fatty matters and epidermis and to open up fiber structure. The process of unhairing depends upon the phenomenon of destroying or loosening and removal of hair by chemical and mechanical means. The keratinous material (hair, hair root, epidermis etc.) are eliminated from the pelts conventionally with the mixture of sulphides (Na_2S, NaHS) and lime which contribute to the high Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Dissolved Solid (TDS) etc. in the tannery effluent. The study showed that significant amount of reduction in pollution concerning parameters like COD, BOD, suspended solids, Nitrogen and Sulphide in tannery waste water due to use of thio compound as unhairing agent. This reduction is explained by the fact that thio compound is non toxic. The intact hair is also collected as new raw material for fertilizer rather than being discharged with the effluent. The strength and organoleptic properties, chromium content and SEM analysis of the leather processed using thio compound, indicates that the quality of leather is also considerably improved.

This paper is focused on development of eco-friendly hair saving unhairing method using organic thio compound as an unhairing agent and to reduce the use of lime and sulphide in liming operation. It also discusses about the quality of leather and discharged liming waste water and compare with the conventional liming.

Keywords: Biological Oxygen Demand (BOD), Chemical oxygen demand (COD), Hair-save, Immunization of keratin, Liming, Scanning electron micrograph, Unhairing.

1 Introduction

HE leather processing involves various operations in a cascade manner form raw hide to crust leather. The complete leather manufacturing process is divided into three fundamental stages: beam house or pre-tanning, tanning and post tanning. The beam house stages comprise of soaking, liming, unhairing, deliming, bating and pickling.

Soaking [1], [2] is the first pre-tanning operation for treatment of hides and skins with water to clean and rehydrate as green condition. In this stage hides and skins are washed and soaked with surfactants and anti-microbial compounds before further processing.

The soaked hides and skins are treated with lime and sodium sulphide mixture which gives desired swelling of collagen structure to open up the fiber bundle. The quality of ultimate finished leather largely depends on this operation.

A common industrial practice of unhairing and liming is one step. Unhairing is the process of removing hair from the pelt without any damage to them. Once the hair shaft detached from the hide surface it is free to float in the bath and can be separated by filtration (hair saving) or chemically dissolved in the bath itself (destructive unhairing). The hair dissolution implies a much higher organic pollution of the waste water, whereas hair saving technologies needs a proper disposal of the recovered hair.

1.1 Sulphide unhairing and liming

Fig. 1. Action of unhairing agent during Na_2S liming system

sodium sulfide and sodium sulphydrate used are sufficient

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to break down the –S-S- bonds that characterize the keratin structure of the hair [2]. In this process the skins are introduced into a drum or paddle and sodium sulfide, sulphydrate and lime are added. The destruction of the hair results rapidly and within (half an hour) the hair loses most of its fiber structure. The hair destruction system is quite common in leather process for all type of production.

In alkaline solution the keratin –S-S- bond may be broken according to the following equation [1], [5].

$$R\text{-}S\text{-}S\text{-}R + 2Na_2S = 2R\text{-}SNa + Na_2S_2$$

1.2 Hair saving unhairing

The hair saving process involves [6], [7] the introduction of enzymes with lime in a balanced system with careful attention to temperature and concentrations. Removal of hair takes place by hydrolysis of the soft proteins in the hair follicle. The thio-compounds have been used in various industrial applications. It has the capacity to cleavage [8] the cystine molecule of keratin protein.

Here the principle of liming by immunization [3], [9], [10] is generally been used. The soaked hides /skins are pretreated in the drum with an alkali like calcium hydrate. Under the influence of alkali the cystine of the hair transforms into lanthiamine, which can no longer hydrolyzed by reduction (immunization). The subsequent addition of sodium sulphide, for example causes a reduction only in unimmunized hair roots which leads to hair loosening. The comparatively well preserved hair can then be recovered by means of a screen [11], [12].

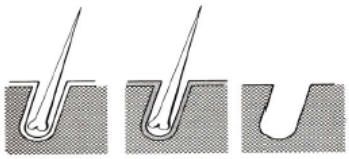

Fig. 2. Action of unhairing agent during a hair-saving system

1.3 The aim of this study

1. Application of organic thio compound as an unhairing agent to reduce the use of lime and sulphide in liming operation.
2. To evaluate the wet blue and leather quality.
3. To study the effect of discharge liming water comparison with conventional liming.

2 Materials and Methods

2.1 Raw hide

In the preliminary trials 8 pieces of raw cow hides (each approximately 2 Kgs) were taken and soaked conventionally. The concentration of thio organic compound was varied over the range of 0.5-2.0 with 1% lime in the liming operation. All the chemicals percentages were based on salt weight and are commercial grade except the organic thiol compound which was prepared from reagent grades chemicals. In this work (no of) domestic cow sides weight ranges 6-7 Kgs were selected as raw materials.

2.2 Shrinkage temperature analysis

The shrinkage temperature (Ts) of samples, which is a measure of hydrothermal stability of leather, was determined [13] using a Theis shrinkage meter. Each value reported is an average of three experiments.

2.3 Analysis of hide substance content of leather samples

Total nitrogen contents of leather samples were measured [13] according to GB4689 using BUCHI-339 device. Hide substance contents of leather samples were calculated by multiplying the measured total nitrogen contents of leather samples by 5.62.

2.4 Mechanical properties of crust leather samples

Mechanical properties such as tensile strength, elongation, tear strength and grain crack strength were measured [13] according to standard procedures. Each value reported was an average of four (2 along the backbone, 2 across the backbone) measurements.

2.5 Analysis of waste water

Waste water was collected from dehairing process for the testing of total solids (TS), chemical oxygen demand (COD) and biochemical oxygen demand (BOD) and sulphide by standard method [9]. The results reported were average values of three experiments for each sample.

2.6 Analysis of total nitrogen in waste water

Total nitrogen contents of waste water collected from dehairing process to deliming process was measured [9] according to standard method.

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2.7 Microscopic studies

Experimental as well as control leather surfaces were examined for variations in surface characteristics using FEI, Quanta 200 Scanning Electron Microscope [9], [14]. Samples from crust leather sides were directly cut into specimens with uniform thickness without any pretreatment.

2.8 Visual assessment

The experiment and control leathers were assessed for organoleptic properties by three experts.

3 Results and Discussion

Recipe 1: Conventional soak and lime process for wet salted hides (% based on wet salt weight.)

Process	%	Chemical	Temperature	Time in minutes	pH
Dirt soak	150	Water	28°C	30-60	
Drain Soak	 120 0.5 0.25 1.0	 Water Sodium carbonate Non-ionic emulsifier Enzymatic soaking agent	28°C	 240-360	 9.5-10.5
Drain Wash Drain	 100	 Water	 26°C	 10	
Lime	100 1.0 1.0	Water Liming auxiliary Sodium hydrosulphide 72%	26°C	 45	
	1.0 1.0 0.2	Lime Sodium sulphide 62% Non-ionic emulsifier		 60	
	50	water	26°C		
	2.0	lime		30	
Run on automatic-stop 50 mins/run 10 mins for 12-14 hours					
Drain Wash Drain	 100	 water	 26°C	 15	
Drain Wash Drain	 100	 water	 26°C	 10	
Take out for fleshing					

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Recipe 2: Hair saving and lime process using organic thio compound for wet salted hide (%based on wet salt weight.)

Process	%	Chemical	Temp.	Time in minutes	pH
Dirt wash	**150**	**Water**	**28⁰C**	**30-60**	
Drain Soak	 120 0.5 0.25 1.0	 Water Sodium carbonate Non-ionic emulsifier Enzymatic soaking agent	 28⁰C	 240-360	 9.5-10.5
Drain Wash Drain	 100	 Water	 26⁰C	 10	
Immunise	120 1.2 1.5	Water Thio organic compound lime	26⁰C	 60	
Hair release	1.0 0.2	Sodium hydrosulphide 72% Non-ionic emulsifier		 20	
				20 stop 90 with screening of hair	
Lime	30 1.5 0.5	water lime Sodium sulphide 62%	26⁰C	 30	
Run overnight for (Run 10 mins, stop 50 mins) for 12-14 hours					
Drain Wash	 100	 water	 26⁰C	 10	
Drain Wash	 100	 water	 26⁰C	 10	
Drain and take out for fleshing					

Table 1: Unhairing at different dosage of thio compound

	Condition of limed pelt	
To be detected lime thio compound dosage (% on salted weight)	Unhairing degree	Scud
0.5	No loosening of hair	Present
1.0	Moderate loosening of hair	Present
1.5	Good loosening of hair	Presence is less
2.0	Excellent Loosening of hair	Clear pelt

Average value of two experiments

3.1 Preliminary unhairing

Preliminary unhairing experiments were performed on laboratory scale. Satisfactory unhairing occurred at 2% concentration. The result of unhairing trials are tabulated in table-1.

From the visual observation during unhairing it was quite comfortable to assess the extent of hair and scud removal.

Unhairing studies conducted at the pilot plant scale on three match sides that unhaired using thio compound (recipe 2.) were unhaired using thio compound with three corresponding matched hide sides for the control (recip 1.)

The processes were conducted by stainless steel laboratory drums rotating in temperature controlled bath.

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The half sides were processed to wet blue and samples were collected for shrinkage temperature and determination of chrome content. The remaining half sides then tanned to crust leather and samples were collected for physical testing.

In recipe-1, 3.5% sodium sulphide and 4% lime are used. A solution of sodium sulphide alone with admixture with lime is a strong unhairing agent. It is reported that when lime alone is used, it requires longer time to cause hair removal. Unhairing effect of sodium sulphide is maximum when SH and OH ions in the solution are present equal quantities. It is also reported only 0.6%Na_2S is required for a hair burn process. In practice much higher amount of sodium sulphide are added. The main reason for this is the fact. The rate of unhairing is on the concentration of sulphide ions.

$$2Na_2S + 2\ H_2O = 2\ NaHS + 2\ NaOH$$

$$2NaSH + Ca\ (OH)_2 = 2NaOH + Ca(SH)_2$$

Here sodium sulphydrate act as an unhairing agent.

Total period in conventional liming was about 20-22 hours from the beginning. Next morning, drained away the liquor and run the drum 20 minutes for pulping out the hair. Then rinsed in water, unhaired and fleshed.

When the keratin protein in the hair cannot be chemically degraded and solubilized, it is said that hair is immunized and that such resistance results from the immunization phenomenon.

In recipe 2., the sodium hydrosulphide was used to replace the sodium sulphide partialy and this reduces the alkalinity available from the dissociation of sodium sulphide in water. Thio compound were used as reducing agent (HS-CH_2-CO_2-H).

The soaked hides were pretreated in the drum with a lime and thio compound. Under the influence of lime, the cystine of the hair transforms into lanthionine, which can no longer hydrolysed by reduction. In alkaline solution, the keratine –S-S- bond may be broken according to the following equation [4].

$$R\text{-}S\text{-}S\text{-}R + H_2O = R\text{-}SOH + R\text{-}SH$$

The subsequent addition of sodium sulphide causes reduction only unimmunized hair roots, which leads to hair loosening. Total 1.5% sodium sulphide was used in this experimental trial.

At pH 9.5-10.5, the thio compound spread as far as the root of the hair and started to hydrolyse the pre-keratin of the hair root. The pH value rose to 12.0-12.5 with the addition of lime and the reducing activity of thio compound consequently increases. Under the influence of lime the immunization of the hair shaft was observed within 90 minutes.

At this point, sodium sulphydrate was added which further reduced and hydrolysed the pre-keratin. The well preserved hair was recovered by screening.

In this work sulphide consumption was less in the developed process (recipe 2.) than the conventional recipe 1. The procedure can be carried out in a drum or paddle and also possible to collect hair.

3.1 Standardization of unhairing process

8 pcs of raw cow hide (each approximately 2 Kgs) were soaked. The concentration of thio organic compound was varied over the range of 0.5-2.0 with 1% lime. All the chemical percentages were based on soaked weight. The unhairing efficiency for each trial is given in table 1.

Table 2: Physical and chemical action on prepared wet blue

Test	Thio-compound unhairing	Sulphide unhairing
% Increase in weight	55	45
% Cr_2O_3	3.3	3.5
% Wrinkles	Less prominent	Prominent
Shrinkage Temperature	102^{0}C	100^{0}C

The hair-saving unhairing process was evaluated by measuring % weight change and the degree of wrinkling. The % Cr_2O_3 in the resulting wet blue leather was also determined as it is shown in the table 2. Both the shrinkage temperature and chrome content values are found considerably improved.

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Table 3: Physical-Mechanical properties of crust leather

Test	Thio compound unhairing	Sulfide unhairing
Tearing load (N)	172	165
Grain distention (mm)	7.6	8.3
Shrinkage temperature (^{0}C)	101	101
Tensile strength (N/mm^2)	24.32	22.43
Elongation (%)	35.52	39.51

Table 3 displays the physical tests, tear strength, tensile strength for both processed leather. The thio compound treated leather can be used for shoe upper since the value for tearing load and grain cracking was more than 120 N and 7.0 mm for distension.

The hair-saving unhairing process showed good physical-mechanical properties, comparable with those of the traditional. In fact the tear strength of the experimental leather was slightly higher as compared to corresponding control leather.

Table 4: Technical properties of crust leather

Technical properties	Thio-compound unhairing	Sulphide unhairing
Roundness	5	5
Fullness	5	4
Softness	5	4

The unhairing quality was evaluated by the appearance of the hair root on the leather by the roundness and softness after being staked.

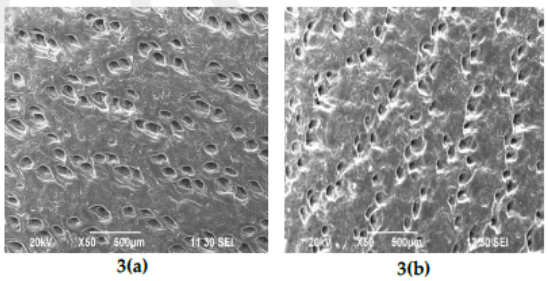
The assessment results of the technical properties of hair saving unhairing system incomparision with the traditional sulphide unhairing system are reported in Table 4. A conventional scale of grades ranging from 1 (worst performance) to 5(least performance) has been used. It may be observed that the crust leather obtained by hair saving unhairing system and traditional sulphide unhairing system show quite similar technical properties.

Table 5: Comparison between the Polluting charge of (unhairing Exhaust Bath of thio-compound and sulfide)

Test parameter	Hair saving (thio compound)	Hair pulping (sulphide unhairing)
COD mg O_2/l	22,700	47,900
BOD mg O_2/l	12000	31,000
N_{TK} mg/l	1800	2560
TSS g/l	4.7	6.2
S^2 mg/l	447	771

The beam house phases, particularly the liming process, make up the most constituent part of total pollution produced due to their considerable contribution in the chemical and biochemical demand and total nitrogen and suspended solid.

Hair-save unhairing process has been applied that significantly reduce (25-30%) the BOD, COD and the total solids in the liming discharge. The result also shows the reduction of the sodium sulphide used with the consequent reduction of nitrogen. This is due to the presence of pollutants in low amounts of waste water in the case of developed hair-save unhairing system.



3(a) 3(b)

Figure: 3(a)- Scanning electron micrograph of a control and 3(b)- experimental crust leather showing the grain surface at magnification of X50

Scanning electron micrograph of crust leather samples processed through conventional unhairing and hair save unhairing process, showing the grain surface at a magnification of X50 are given in Fig. 3(a) and 3(b) respectively. It is seen that the hair pores of experimental samples appears to be much cleaner than control. This indicates that, hair save unhairing system able to perform unhairing without trace of hair. These observations are consistent with the conclusion mentioned above.

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Conclusion

The possibility of unhairing using thio compound has been explored in this study. The use of thio compound has been found as excellent unhairing agent. The grain character and the strength properties of processed leather have been found to be good when composed to experimental leather. It is observed that hair was recovered easily by screening. This study was carried out in drum and the condition needs to modify for collection of hair. This brings about a significant reduction of the sodium sulphide and suspended solids with the consequent reduction in odour (H_2S gas).

The global concern for cleaner leather production has led tanneries to reduce the elimination of toxicity in their effluents. The possible application of hair saving unhairing process using thio compound confirmed.

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