COIR FIBRES : MODIFICATIONS, CHARACTERIZATION AND APPLICATION IN FIBROUS COMPOSITES

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CERTIFICATE

This is to certify that the thesis entitled "COIR FIBRES: MODIFICATIONS, CHARACTERIZATION AND APPLICATION IN FIBROUS COMPOSITES" submitted by Manika Varma to the Indian Institute of Technology, Delhi for the award of the degree of Doctor of Philosophy is a record of bonafide research work carried out by her. Manika Varma has worked under our guidance and supervision and has fulfilled the requirements for the submission of the thesis, which to our knowledge has reached the requisite standard.

The results contained in this thesis have not been submitted, in part or full, to any other University or Institute for the award of any degree or diploma.

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ABSTRACT

Characterisation, modifications and the potential applications of coir (Cocos nucifera), a naturally occuring ligno-cellulosic fibre, in fibrous composites has been investigated in the present work. Three varieties of available coir fibres (bristle, mattress and decorticated) were characterised. The elemental analysis of these varieties of coir fibres showed similar carbon content (47±0.7%) and hydrogen content (5.27±0.9%). Characteristic absorption peaks of cellulose and lignin, the major components of coir fibres were observed in the FT-IR spectra of these fibres. Percentage crystallinity index determined by X-ray diffraction technique was found to be comparable (44.6±0.4%). Except for density of bristle coir fibres, which was found to be highest (1.401 gcm) no significant difference was observed in the structure of the three varieties of coir fibres. Morphology as studied by SEM revealed an uneven surface and presence of globular protrusions embedded on the fibre surface. Mechanical properties of these fibres were comparable (tenacity was found to be 2.2±0.1 g/denier, initial modulus was 36±2g/denier and elongation-at-break was 30±2%).

Decoricated coir fibres showed lower moisture regain at humidities ranging from 20-100%, while mattress showed much higher moisture regain at humidities above 75% RH, compared to bristle coir fibres. A moisture regain of 9% at 66% RH was observed for bristle coir fibres.

In order to improve (a) the wettability of the fibres (b) reduce the moisture regain (c) improve the interfacial bonding with unsaturated polyester resin (USP), surface modifications of these fibres was attempted.

Coir fibres were treated with sodium hydroxide at room temperature. Effect of concentration (5-10%) and duration of alkali treatment (1-24h) was investigated. Alkali brought about loss in weight of coir fibres. A loss of 9% was observed upon treatment with 10% alkali for 4h. Carbon content decreased while an increase was observed in hydrogen content. Mattress and decorticated fibres showed similar changes. In the FT-IR spectra of treated fibres, the absorption peak at 1740 cm^{-1} disappeared thereby indicating the rupture of alkali sensitive ester linkages. Density of the fibres increased by $5\pm2\%$, while an increase of $10\pm3\%$ was observed in % crystallinity index. Initial modulus increased by about 16% for bristle coir fibres, while a slight reduction was observed in tenacity and elongation-at-break. Appearance of several voids on fibre surface was revealed by SEM.

Hydrochloric and acetic acid treatment of coir fibres at room temperature was carried out and the effect of reaction time (1-4h) and concentration (5-15%) of the acids was investigated. Approximately 8% loss in weight was observed upon treatment with 10% HCl for 4h. No significant change was observed in elemental analysis. Density of acetic acid treated fibres, increased slightly while that of HCl treated fibres decreased. % crystallinity index increased while Hermans orientation factor decreased upon HCl treatment. Mechanical properties deteriorated and moisture regain increased on acid treatment of fibres.

In order to reduce the concentration of hydroxyl group in these fibres, reaction with isopropyl triisostearoyl titanate, (TTS), tolylene diisocyanate (TDI), γ-methacryloxy propyl trimethoxy silane (MAPS), dichloro methyl vinyl silane (DMVS) and sebacoyl chloride (SC) was investigated. Effect of reaction parameters (duration, concentration of reagent, temperature and solvent) was investigated.

Treatment with TTS was carried in benzene solution both at room temperature as well as at 78 ^{o}C. A decrease in elongation-at-break and a significant reduction in moisture regain was observed on TTS treatment of coir fibres. Deposition of titanate coupling agent on coir fibres was confirmed by the appearance of an absorption band at 810 cm^{-1} in the IR spectrum of the treated fibres. No significant change was observed in elemental analysis, density and % crystallinity index of the treated fibres indicating that reaction was confined to surface only.

MAPS treatment of fibres did not alter the structure or the mechanical properties of the fibres. However, upon DMVS and SC treatment, mechanical properties deteriorated and moisture regain increased significantly. This indicated the degradation of the fibres by the HCl liberated during the reaction.

Reaction of TDI with active functional groups in coir fibres was investigated in several solvents (eg. benzene, dichloroethane, dimethyl sulfoxide, etc.,). Polarity of the solvent affected the reaction and an increase in nitrogen content of the fibres from 0.15% to 0.35% was observed in DMSO solution of TDI,indicating that TDI had very low reactivity. Carbon and hydrogen content was marginally affected. Density and %

crystallinity index of the treated fibres increased. Moisture content of the treated fibres decreased at humidities below 75% RH, while a slight increase was observed above it.

Bristle coir fibres were also coated with a dilute solution of USP(in ethyl methyl ketone). New absorption peaks were observed at 710 cm^{-1}, 745cm^{-1} and 800 cm^{-1} and an increase in the intensity of absorption peak at 1740 cm^{-1} confirmed the deposition of USP in the fibres. Significant lowering in moisture regain was observed at all relative humidities (eg. a decrease of 23%) was observed at 66% RH). SEM of the surface confirmed the uniform deposition of a thin coat on the fibre surface.

Mechanical properties of neat USP sheets were also evaluated. Dough moulding compounds were fabricated with chopped bristle coir fibres (6mm long), chopped glass fibres, $CaCO_3$ as a filler and USP as a binder, using compression moulding technique at 100°C . Partial replacement of glass fibres by coir fibres was possible and compounds with good properties (tensile modulus of $34.9x10^3$ psi) could be obtained.

Hybrid composites made with chopped bristle coir fibres as core and polyester compatible glass fabric (Fibre glass Pilkington) on the outside and unsaturated polyester resin as matrix, were fabricated using a compression moulding machine. Mechanical properties were found to vary as a function of fibre length and fibre weight fraction. In comparison to neat polyester sheets 54% improvement in flexural strength and 125% improvement in flexural modulus was observed by using 20% (w/w) of coir fibres (1 cm long). Composites made with coir fibre mat showed similar flexural strength while flexural modulus was found to be lower.

Composites fabricated with alkali, titanate and resin treated fibres showed enhancement in interlaminar shear strength and Izod impact strength. Flexural properties also showed an improvement.

Thermal properties of untreated/chemically treated fibres and of cured resin were studied by thermogravimetry. Untreated and decorticated coir fibres showed step weight loss. Lowering of moisture content upon TTS and resin treatment was confirmed by TGA by lowering of % weight loss in 40 -150 ^{0}C region. For untreated mattress, alkali and DMSO treated coir fibres a single step weight loss was observed. Desalted bristle coir fibres showed a higher thermal stability. Similar increase was also observed for alkali and acid treated coir fibres.

Photostability of the three varieties of coir fibres was evaluated by exposing them to UV radiation for 100h. About 40% reduction in mechanical properties was observed on irradiation. Fibres treated with sodium bisulfite retained 75% of their strength.

Neat USP resin sheets stabilised by the addition of UV light stabilisers (Tinuvin 770 and 327) showed better retention of properties when Tinuvin 770 was used.

The effect of subjecting the composites to humid conditions RH (40-90%) on the mechanical properties was also evaluated. Composites made with chopped coir fibres showed only 76% retention in flexural strength and 78 % retention in flexural modulus at 84 % RH, while composites made with coir fibre mat showed a 70% and 75% retention respectively.

Composites fabricated with chemically treated coir fibres retained the mechanical properties at all humidities. Maximum retention for chopped coir fibre composites was observed for TTS treated fibre composites, while 10% resin treated coir fibre mat composites seemed most effective in case of composites made with coir fibre mat.

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