PREPARATION AND CHARACTERIZATION OF LOW DENSITY POLYETHYLENE-BASED COMPOSITE MATERIALS CONTAINING RICE-STRAW AS A FILLER

by

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DECLARATION

I hereby declare that this submission is a result of a work carried out by me and to the best of my knowledge, it contain no material previously written or published by another person nor material which has been accepted for the award of any degree or acceptable qualification of a university, or other Institute of higher learning, except where the due reference to the material is made.

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December 21 2004

To the best of my knowledge, the above particulars are correct.

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(Dr. B A J K Premachandra)

ABSTRACT

This thesis consists of four chapters. Chapter one contains an introductory part including scope and the objectives of the research. Since the present research is divided in to three major parts, chapter two, three and four are arranged accordingly. Each chapter includes an introduction discussing the relevant literature, the experimental work, results and discussions and finally, the conclusions drawn. The contents of this thesis can be best summarized as follows.

Preparation and characterization of composite materials containing LDPE and untreated rice-straw powder

A series of Low Density Polyethylene (LDPE)/straw composites containing different amount of straw (as wt %) were prepared using rice-straw powder having three different particle sizes (50 μm, 90 μm and 250 μm). Mixing was done by melt-mixing technique, where LDPE and straw powder were mixed in a Brabender plasti-corder, which was operating at 170°C temperature and 60 rpm. The composites were studied by using Fourier Transform Infra-red spectroscopy (FTIR). The failure modes, tensile properties, water absorption, biodegradation and weatherability were investigated as a function of the weight percentage as well as particle size of straw filler in the composite. It was found that the incorporation of straw into LDPE matrix has reduced the ductility of the composite. The mechanical properties of the composites, especially the tensile strength and elongation at break were significantly low compared to those of neat LDPE. A significant improvement in modulus was shown by the composites. It was found that the tensile properties were depended on the amount and particle size of the straw in a composite.

Biodegradability of cellulose component in the rice-straw and LDPE-straw composites, after exposure to cellulase enzyme solution, was assessed by weight loss measurements. It was found that rice-straw sample is readily biodegradable but degradability of the composite samples was not significantly affected by the cellulase enzyme.

The extent of degradation after the weathering process was assessed by the loss of tensile strength measurements of the composites after incubation of the samples in a weather meter, which was at 70^0 C and continuous UV and moisture cycles for five days. It was found that the degradability of the composite samples depends on the amount of the rice-straw in a sample.

Preparation and characterization of maleic anhydride grafted LDPE and composite materials with maleic anhydride grafted LDPE and untreated rice straw

A series of maleic anhydride grafted LDPE samples with different wt%'s of maleic anhydride and dicumyl peroxide were prepared and studied. The maleic grafted LDPE samples were prepared by melt free radical grafting method, where the grafting reaction of LDPE was carried out with the free radical initiator (Dicumyl peroxide) in a Bra-bender PL2000 plasti-corder operating at 170 ^{o} C. Fourier transform infrared (FTIR) spectroscopy confirmed the grafting of maleic anhydride on LDPE backbone. Melt viscosity measurements and tensile measurements of grafted LDPE samples confirmed the unwanted cross- link formation during the grafting reaction.

A series of composites with maleic grafted LDPE and different composition of untreated rice-straw were prepared using simultaneous grafting and mixing technique. In this regard LDPE, maleic anhydride, dicumyl peroxide and rice-straw were fed in to the hot plasti-corder operating at 170^o C, where melt free radical grafting reaction as well as melt mixing of straw filler with LDPE were occurred. Fourier transform infrared (FTIR) spectroscopy confirmed the formation of new interface interaction (ester bond) between the rice-straw and maleic grafted LDPE. The failure modes, mechanical properties, rheological properties, water absorption, biodegradation and weatherability were investigated with respect to the weight percentage of the straw as well as particle size of the straw in the composite sample. Improved mechanical properties, especially tensile strength and modulus were also evidenced the compatibility and interface interaction in the maleated LDPE-straw composites. It was found that the tensile and modulus values of maleated LDPE-straw composites having smaller particle size and higher filling level of rice-straw to be higher due to the formation of more interactions at the interface. According to the melt viscosity and shear rate analysis, higher melt viscosity was shown by the maleated LDPE-straw composites due to the undesirable cross-links formed in the maleation process. Compared to the LDPE-straw composites, the corresponding maleated LDPE-straw composites have shown higher water absorption. As in the case of LDPE-straw composite these composites also have not shown any weight loss after the digestion with cellulase enzyme but have shown a considerable degradability after the weathering process.

Preparation and characterization of composite materials with maleated LDPE and treated rice-straw

Using simultaneous grafting and mixing technique, another series of composites were prepared with maleated LDPE and treated rice-straw powder. In order to remove lignin and other waxy substances, rice-straw was subjected to steam explosion and hot alkyl treatments. By the chemical analysis results it was found that most of the lignin and other waxy substances have removed from the straw surface. Fourier transform infrared (FTIR) spectroscopic results also confirmed the removal of lignin and thereby increase of wt% of cellulose in rice-straw. Further FTIR analysis of the composite sample more clearly showed the formation of interface ester linkages between the treated straw and maleated LDPE.

Similar to the chapter three, different property analysis such as mechanical, water absorption, enzymatic digestion and weatherability were carried out with respect to the weight percentage and particle size of the straw filler. Compared to the LDPE-straw composites and maleated LDPE-untreated straw composites, significant improvement in the mechanical properties were resulted in the maleated LDPE-treated straw composites. It was also evidenced that the removal of lignin by the treatment processes has enhanced the interface interaction of maleated LDPE-straw system. Further it was found that filler properties such as the amount of straw filler in the composite and the particle size of straw also govern the mechanical properties of the composites. Providing more surface area and more OH groups to form ester bonds, composites having smaller size and higher amount of straw have shown higher tensile and modulus values. Also in this series the percentage elongation properties were significantly reduced with the introduction of the treated straw in to

LDPE but the reduction is significantly lower than the composites with maleated LDPE-untreated straw. The extent of the biodegradability of the composites with cellulase enzyme was also analyzed by the weight loss measurements. As in the above two composite series maleated-treated composites also have not shown significant digestion with the cellulase enzyme.

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CCl_4	. Carbon tetra Chloride
$CaCO_3$	- Calcium Carbonate
DCP	- Dicumyl Peroxide
DP	- Degree of Polymerization
FTIR	- Fourier Transform Infra- Red
KBr	- Potasium Bromide
HDPE	- High Density Polyethylene
LDPE/PE	- Low Density Polyethylene
MA	- Maleic Anhydride
MPa	- Mega Pascal
MPETS	- Maleated LDPE/Treated straw composite
MPES	- Maleated LDPE/Un Treated straw composite
NaOH	- Sodium hydroxide
PES	- LDPE/Un Treated rice-straw composite
PMPPIC	- Poly[methylene (poly (phenyl isocyanate))]
PP	- Polypropylene
PS	- Polystyrene
PVC	- Polyvinyl Chloride
rpm	- Rounds per minutes
TAPPI	- Technical Association for Pulp And Paper Industry
TDIC	- Toluene2, 4- diisocyanate
%T	- Percentage transmittance
UV	- Ultraviolet radiation
Wt %	- Weight percentage
3-D	- Three dimensional