



Studies of Chemical Properties of Rice Husk Polypropylene Composites

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Rice husk polypropylene composites were prepared by the process of extrusion and compression molding using maleic anhydride polypropylene as compatibilizer. Change in appearance of rice husk polypropylene composites was noticed when dipped in H₂O, NaOH, NH₃, HCl. Significant swelling was observed in rice husk polypropylene samples when dipped in NaOH whereas rice husk polypropylene samples showed surface roughness when dipped in HCl. Tensile strength of rice husk polypropylene composites decreased after dipping in all chemicals except in NH₃, samples dipped in ammonia have better tensile strength, showing the suitability in ammonia environment.

Keywords: Rice husk, Ammonia, Compatibilizer, Tensile strength, Swelling.

INTRODUCTION

Yang *et al.*¹ studied that commodity plastics such as polypropylene (PP) can be reinforced with rice husk for development of thermoplastic composites. The addition of rice husk in polypropylene decreased mechanical properties such as tensile and impact strength due to poor interfacial bonding between polypropylene and rice husk. There was improvement in tensile modulus of composites by addition of rice husk. Use of compatibilizer enhanced tensile strength without improving impact properties as studied by Yang *et al.*² Use of higher temperature lowered the tensile strength and modulus of composites. Fuad *et al.*³ studied the effect of coupling agents such as titanate (LICA 38), zirconate (NZ 44) and silane (PROSIL 2020 and PROSIL 9234) on rice husk ash (RHA) based polypropylene composites. Lower melt flow index and higher flexural modulus was obtained with higher filler loading. Addition of PROSIL 2020 coupling agent improved tensile strength but higher impact strength was possible with LICA 38 and PROSIL 9234. Addition of zirconate (NZ 44) resulted better stiffness in composites. Natural fibers such as sisal, jute were used with polyester and epoxy matrices for making composites as studied by Mathur⁴. Low cost, durability and reduced moisture absorption are advantageous for building sectors in form of panels, door, roofing sheets, laminate, shuttering. Wood plastic composites (WPC), could be utilized in various applications of automotive industries since these materials have superior strength/stiffness ratio, less abrasive, good acoustic

performance, less fuel consumption, biodegradability, improved passenger safety and shatter proof performance under extreme temperature changes as studied by Ashori⁵.

Blackadder and Poidevin⁶ studied that dissolution of polypropylene in solvent is dependent on polymer and solvent both. Experimental conditions such as temperature and degree of stirring significantly affected the dissolution process. Isotactic polypropylene was immersed in tetrachloro ethylene in temperatures range (368-373 K) and change in molecular weight, crystallinity and morphological behaviour was noticed by Drain *et al.*⁷. Crystalline, amorphous phases and different molecular weight fractions have different solubilities. Scanning electron microscopy (SEM) confirmed dissolution of polypropylene in tetrachloro ethylene. Liu *et al.*⁸ studied grafting of methyl methacrylate (MMA) onto isotactic polypropylene (ipolypropylene) film by using supercritical carbon dioxide (SC CO₂) which acts as solvent and swelling agent both. Grafting level and morphology was controlled by soaking time, pressure and concentrations of methyl methacrylate and benzoyl peroxide. Melting temperature of grafted copolymer reduced as compared to virgin isopolypropylene, studied by DSC. It might be due to lower crystallinity of grafted isopolypropylene with increasing degree of grafting. Polyolefin was used as matrix and wood flour as reinforcing filler, with commercial particle board, medium-density fiber board and solid woods (red pine and birch) as studied by Yang *et al.*⁹. Negligible increase in thickness swelling and water absorption of biocomposites with higher filler loading was noticed, as compared to wood based

composites (particleboard and fiberboard) and solid woods (red pine and birch) by adding compatibilizer. Mechanical properties of composites decreased with filler loading, still bio composites have good strength for interior of bathrooms, wood decks, food packaging. Nouri *et al.*¹⁰ studied chopped rice husk (CRH) based polypropylene composites with maleic anhydride grafted polypropylene as compatibilizer. It was found that flexural strength, tensile and flexural modulus of composites enhanced with CRH loading but elongation at break and energy at break decreased. Water absorption studies showed an increase in diffusion coefficient with chopped rice husk loading. Fávaro *et al.*¹¹ studied the post consumer high density polyethylene (PE) reinforced with rice husk, which was mercerized with NaOH solution and acetylated. Addition of rice husk fibers into PE matrix increased tensile and flexural modulus of composite prepared with 10 wt. % of acetylated rice husk fiber and unmodified polyethylene matrix. Increase in Izod impact strength as compared to pure matrix was also noticed.

EXPERIMENTAL

Following materials were taken for the study: polypropylene, rice husk, compatibilizer and chemicals.

Polypropylene: Polypropylene was procured from M/s Reliance Industries Limited (grade 110 MA), Mumbai, India and was tested for following properties:

- Melt flow index (ASTM D1238): 19.09×10^{-5} kg/s
- Moisture content (ASTM D789): 0.039 %
- Tensile strength (ASTM D638): 29.09×10^6 kg m/s² (29.09 MPa)
- Notched Izod impact strength (ASTM D256): 20.42 kg m/s²
- Hardness (ASTM 2240): 63.75 (Shore D)
- Abrasion resistance (ASTM D4060): 22.12×10^{-6} kg/1000 cycles

Rice husk: It was obtained locally, grinded to fine powder (24 mesh) and following properties were measured:

- Bulk density (ASTM D1895): 104.4 kg/m³
- Moisture content (ASTM D789): 6.8 %
- Ash content (ASTM D2584): 14.4 %

Compatibilizer: Maleic anhydride polypropylene (grade P 406) compatibilizer was supplied by M/S Pluss Polymers Private Limited, Gurgaon, India and percentage addition was 3 % (of total weight of rice husk polypropylene composite) as recommended by supplier.

Chemicals: H₂O, NH₃, HCl and NaOH were used to study chemical properties of rice husk polypropylene composites. NaOH was procured from M/S Central Drug House, New Delhi, India (molecular weight 40, minimum assay 96 %). NH₃ was procured from M/S Ranbaxy Fine Chemicals Limited N. Delhi, India (specific gravity 0.91, molecular weight 17.03 and minimum assay 25 %). HCl was procured from M/S Merck India Limited, Mumbai, India (molecular weight 36.46, minimum assay 35 %).

General procedure: After carrying out the raw material testing, processing of composite was done on single screw extruder and compression molding machine. Before processing on compression molding, raw materials were passed through single screw extruder to obtain granules of uniform size and composition.

Single screw extruder: Single screw extruder used was supplied by M/S Lakshmi Industries Ludhiana, India with following specifications.

L/D ratio 20:1 and compression ratio 3:1

Temperature setting in extruder: 423, 453, 488, 483 K

Machine was made on and allowed to reach the set temperature of 483 K. After attaining required temperature, raw materials (polypropylene, rice husk and compatibilizer) were fed from hopper into the barrel. Raw materials were sheared between screw and barrel, finally forced by screw towards die. Molten plastic was passed through the screen pack which removes contaminants present in melt, breaker plate was used to reinforce screen to encounter excessive pressure build up. Plastic strands were produced by die and passed through water bath for cooling. Granules of uniform size (2×10^{-3} m diameter approx.) were obtained and used as raw material on compression molding process for making composite sheet.

Compression molding process: Compression molding machine was supplied by M/S Enkay, New Delhi, India. Mold was rectangular in shape to produce composite sheet of $155 \times 155 \times 10^{-6}$ m².

Compression molding machine was switched on and required temperature was allowed to attain. Mold halves were cleaned and silicon was applied on the mold surfaces. Plastic granules were kept in mold halves and mold was clamped in semi automatic compression molding machine. Following are optimized conditions for getting good quality sheet of rice husk polypropylene composite. Processing temperature: 473 K. Clamping pressure: 590×10^4 kg/m². Heating time: 900 s. Cooling time: 1800 s.

After completion of heating cycle, mold was cooled by circulating water. Mold was taken out from machine and two halves were opened. Rice husk polypropylene composite sheet was obtained for analysis of chemical properties.

Detection (testing) methods: After preparing the RHPP sheet, standard size of samples were cut to check the following properties *i.e.*, swelling, chemical resistance and tensile strength.

Swelling (percentage weight gain ASTM D471): Rice husk polypropylene sample of size $25 \times 50 \times 3 \times 10^{-9}$ m³ was cut from the sheet, weighed accurately. These samples were dipped in HCl, H₂O, NaOH, NH₃, kept in glass containers. Samples were removed after 3.6×10^3 s and physical appearance was checked. Weight of samples after dipping was taken to calculate swelling (percentage weight gain) as per eqn. 1.

$$\text{Weight gain (\%)} = \left[\frac{(\text{Final weight} - \text{Initial weight})}{\text{Initial weight}} \right] \times 100 \quad (1)$$

Chemical resistance of plastics (ASTM D543): In order to analyze the effect of chemicals on rice husk polypropylene samples, same experiment was performed but samples were dipped in acids and bases for a week. Sample dipped in HCl was removed just after 3 days due to generation of excessive fumes and appearance of samples dipped in all chemicals was checked.

Tensile strength (ASTM D638): Machine used for tensile testing was LR100 K Plus (M/S Lloyd, England) with 100,000 kgm/s² capacity. In the above test, gauge length was fixed at 50×10^{-3} m and testing speed was set at 83×10^{-5} m/s. Sample size was $50 \times 12.7 \times 3 \text{ mm} \times 10^{-9} \text{ m}^3$ and clamped between

stationary and movable jaws. Sample start to elongate as machine was started and extensometer gave elongation reading on stretching. At complete failure, load and deflection was noted to calculate tensile strength as per eqn. 2.

$$\text{Tensile strength} = \frac{\text{Force}}{\text{Cross sectional area}} \quad (2)$$

RESULTS AND DISCUSSION

Effect of chemicals on physical appearance of rice husk polypropylene samples: Effect of dipping of rice husk polypropylene samples in various chemicals is illustrated in Table-1. Rice husk polypropylene samples dipped in HCl have dull surface which might be due to its corrosive nature. Significant swelling is noticed in rice husk polypropylene samples dipped in NaOH due to its hygroscopic nature. No noticeable change in the appearance of rice husk polypropylene samples is observed when dipped in water and ammonia.

TABLE-1
EFFECT OF CHEMICALS ON PHYSICAL APPEARANCE
OF RICE HUSK POLYPROPYLENE SAMPLES

S. no.	Chemicals used	Physical appearance
1	HCl	Dull surface
2	H ₂ O	No change
3	NaOH	Swelling
4	NH ₃	No change

Swelling (percentage weight gain) of rice husk polypropylene samples: Percentage weight gain calculated as per eqn. 1 is reported in Table-2. Swelling is noticed in rice husk polypropylene samples dipped in all chemicals, it might be due to the presence of amorphous portions in plastics which allow penetration of these chemicals. Rice husk polypropylene samples dipped in NaOH have maximum swelling due to its hygroscopic nature.

TABLE-2
EFFECT OF CHEMICALS ON SWELLING AND TENSILE
STRENGTH OF RICE HUSK POLYPROPYLENE SAMPLES

S. no.	Chemicals used	Swelling (%)	Tensile strength after dipping (MPa)
1	RHPP original	-	23.62 (without dipping)
2	RHPP in HCl	0.42	14.02
3	RHPP in H ₂ O	0.65	21.75
4	RHPP in NaOH	3.1	12.32
5	RHPP in NH ₃	0.46	28.30

Effect of chemicals on tensile strength of rice husk polypropylene samples: Result for tensile strength before and after dipping is reported in Fig. 1. There is decrease in tensile strength of rice husk polypropylene samples dipped in all chemicals except in ammonia. Significant reduction in tensile strength of rice husk polypropylene samples (dipped in HCl and NaOH) result due to their strong acidic and basic nature. Decrease in tensile strength is due to degradation of fibers/matrix interfacial bond as absorption of chemicals by the matrix and interfaces, which reducing the connectivity of phases as studied by Roberts¹². Samples dipped in H₂O, do not show much change in tensile strength. For samples dipped in ammonia,

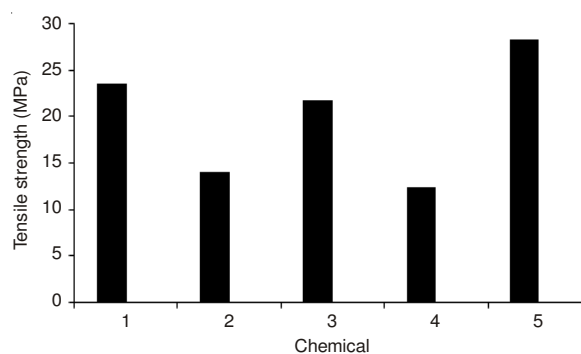


Fig. 1. Effect of chemicals on tensile strength of rice husk polypropylene composites

tensile strength is improved from 23.62 to 28.30 $\times 10^6$ kg m/s². Orientation/crystallization of rice husk polypropylene composites in liquid ammonia have positive effect on the crystallinity of rice husk polypropylene composite resulting better tensile strength as studied by Bagherpour *et al.*¹³.

Conclusion

Noticeable change in the appearance of rice husk polypropylene samples after dipping in chemicals is observed. Rice husk polypropylene samples dipped in HCl have roughness whereas considerable swelling is noticed in rice husk polypropylene samples when dipped in NaOH. Swelling is important for gaskets, hoses and seals which may come in contact with chemicals. Exposure of rice husk polypropylene samples to various chemicals decrease tensile strength except for ammonia. When exposed to ammonia, higher tensile strength is noticed, showing suitability of rice husk polypropylene composites in NH₃ environment. Good chemical properties of rice husk polypropylene composites could be of importance in various applications such as chemical tanks, industrial piping, automotive batteries, lab ware. Present study explores the possibility of rice husk polypropylene composites for various applications of chemical industries.

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