

Mechanical and thermal studies on rose petals using polypropylene bio composites.

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Abstract:

During the recent years, polymeric composite materials are being used in variety of applications due to their high strength and stiffness, light weight and high corrosion resistance. Most of the products are made from non bio degradable fibers, where the problem of disposal arises after the end use; this raised the attention of people for the use of natural, sustainable, biodegradable and renewable resources. The use of various natural reinforcing fibers in thermoplastics with the fibers such as hemp, jute, flax, sisal, and kapok had gained acceptance in commodity plastics and applications of these materials during the past decade has been reported in literature. However, still various fibers and particulate fillers need to be explored. The aim of the present work is to develop polypropylene (PP) composites, using renewable bio resources. Rose petals powder was used as natural reinforcing fillers in this research work. They are relatively inexpensive and abundantly available in nature. The mechanical and thermal studies are carried out to evaluate the effect of filler content on polypropylene. Impact strength also decreases with filler content but improved strength is obtained for maximum filler composite. This project has shown that the composites with maleic anhydride as a coupling agent will be desirable for making house hold products due to their considerable stability and strength properties. The prepared composite samples were characterized with different techniques to ascertain their utility house hold and industrial applications.

Keywords: Polypropylene, agro waste, coupling agent, bio composite, thermoplastic composites.

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I. Introduction

Ligno cellulosic materials have become important as fillers or reinforcements in polymer or ceramic matrices due to their advantages in relation to other inorganic or synthetic materials because of their easily recyclable, minimum requirements, non-abrasive to machinery, stronger performance even with lower weight, impact and shatter resistant. Further they have lower processing energy requirements and a low thermal expansion coefficient; they have a natural appearance, are easily coloured and are low cost, costing less than the base resin. Several ligno cellulosic materials are used as fillers or reinforcement in thermoplastic composites, including fibres of sugarcane, banana, jute, ramie, flax, pineapple, sisal, cotton, heart-of-peach palm sheath, bagasse, sunflower stalk flour, and palm leaf waste, as well as fibres and wood particles of different tree species. Rose petals are one of the most popular flowers in the world. The other developmental traits, there are clear differences between cultivars in their response to temperature. Various parts of plant have many applications, amongst which post fiber application in reinforcing thermo and thermosetting plastics had gained importance in the current decade. Fibers obtained from rose petals are a fiber which has a structure and relatively good mechanical properties. They consist of helically wound cellulose microfibrils in amorphous matrix of lignin and hemicellulose. Lignin are composed of nine carbon units derived from substituted cinnamyl alcohol which is in turn associated with the hemicelluloses and play an important role in the natural decay resistance of the ligno cellulosic material. It can be noted that cellulose is the main constituent of plant fibers followed by hemi-celluloses and lignin interchangeably and pectin respectively. Cellulose present in the plant fiber in turn acts as the reinforcement for lignin, hemi cellulose and pectin. This makes plant fibers exhibit characteristics of a composite material. From the available literature, it was observed that the rose petals fiber possesses good specific strength properties comparable to those of conventional materials like glass fibers. Therefore utilization and application of the cheaper goods in high performance appliance is possible with the help of this composite technology. Alkali treatments have been proven effective in removing impurities from the fiber, decreasing water sorption and enabling mechanical bonding and thereby improving matrix interface interaction. Therefore an effort had been taken to treat the rose petals fibers with sodium hydroxide (6wt %) for the reinforcement in polypropylene matrix and to study their mechanical and thermal properties.

II. Objectives Of The Research Work :

Utilization of biodegradable agricultural waste such as vegetable waste for the development of composites. Choice of polypropylene as the matrix material for preparation of the composites because of its huge commercial availability. To evaluate and report the efficient processing methods and their relationship with varying percentages of filler as reinforcement. To study the characteristics of polypropylene composites in terms of various physical parameters essential for their acceptance as marketable products.

III. Materials And Methods:

The commercially available and industrially important polypropylene was used in the present study as a matrix material is homo-polymer grade (Repol H110)-P2, which were supplied by Reliance Industries. Renewable reinforcing filler used is rose petals. The compatibilizers used were maleic anhydride grafted polypropylene-MAPP (Amplify GR 216) with density of 0.875g/cm³ was obtained from Dow chemicals (Bhimrajka Implex Ltd.).

A. PREPARATION OF FILLER WITHOUT CHEMICAL TREATMENT:

Rose petals were used as such without any modification and with modification by chemical treatment to compare the properties of the prepared composite with respect the virgin matrix.

Figure 1: Rose petals



B. DRYING OF FILLERS

Flowers with yellow petals were picked by hands. Spread under sun light for 3 days, then moved to shadow until they are completely dry. The dried flowers are ground into powder using a food processor to get filler powder.

Figure 2: Drying of fillers in sunlight

DAY 1



Figure:2

DAY 2



Figure:3

DAY 3



Figure:4

C. GRINDING AND SIEVING OF FILLERS :

The dried flowers are ground into powder using a wood grinding machine to get filler powder. The powdered filler are passed into the sieve shaker to get uniform particle size of <75 micro metre



Figure:5

D. PREPARATION OF FILLER WITH CHEMICAL TREATMENT :

10g of rose petals filler in the form of powder was treated with alkaline solution under mild condition by soaking them in 17.5wt% sodium hydroxide solution for 1hr in a glass beaker at room temperature. The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. The resulting mixture was filtered off and the extract obtained is dried and ground again and taken as chemically treated sample.



Figure :6

Chemical treatment of rose petal filler

IV. Compounding:

Polypropylene granules, bio fillers and compatiblizer were pre dried in an air oven at 80°C for 4h and mixed well before blending. The compounding materials were melt blended by directly adding into the feeding zone of twin screw extruder (HAAKE Rheomex OS, PTW16 Thermo Scientific, Germany). Blending was carried out at a temperature range of 210°C, 200°C, 190°C, 180°C and 150°C at a screw speed of 75rpm. The process of mixing with fillers after transfer of polymer materials into mould was completed within 80 seconds to avoid heat loss and to ensure thorough mixing. Compression force of 100kN was applied to the molten polymer mix for about 20 minutes. Mould was cooled by circulating cold water through the columns around the mould.



Figure 7: PP granules



Figure 8: MAPP

| Sample | RPF(Wt%) | RPF(Gms) | PP(Gms) | MAPP(Gms) |
|------------|----------|----------|---------|-----------|
| Neat PP | 0 | 0 | 1000 | 0 |
| URPFPP 5 | 5 | 100 | 1800 | 100 |
| URPFPP 7.5 | 7.5 | 150 | 1750 | 100 |
| URPFPP 10 | 10 | 200 | 1700 | 100 |
| TRPFPP 5 | 5 | 100 | 1800 | 100 |

*PP- PolyPropylene; Rose petals Filler; MAPP – Maleic Anhydride Grafted Poly Propylene U-Untreated, Treated

Table 1: Composition of Samples

Composite samples which were extruded from the mould were in the form of strands, which were further chopped into smaller pellets further characterization and for making test samples according to ASTM standard. Composites of following composition were prepared as given in Table 1. After compounding the samples are taken for the injection moulding.



Figure 9: Extruded RP bio composites

V. Mechanical Testing:

Mechanical Testing were carried out as per American Standard Testing Methods (ASTM). Four tests were performed on bio composites namely tensile strength, impact strength, hardness and flexural test. All the composite samples used for testing mechanical properties were machined into shape by grinding machine according to the ASTM standards and the cut edges were made smooth using sand paper to have a control on the specimen dimension.

A. CONDITIONING:

The test specimens were conditioned at $23\pm 2^{\circ}\text{C}$ and $50\pm 5\%$ relative humidity for 40h prior to the testing.

B. TENSILE TESTING :

Tensile strength is a measurement of the ability of a material to withstand forces that tend to pull it apart and to what extent the material stretches before breaking. Tensile modulus, an indication of the relative stiffness of a material, can be determined from a stress-strain diagram. Tensile properties were studied as per ASTM-D 3039 using Instron testing machine (Model 6025 UK), at 10 mm/minute cross-head speed, using specimen with a width of 25 mm, length of 200mm and thickness of 3mm. A distance of 115mm is kept in between the grips. Five specimens were tested for each sample

C. TENSILE STRENGTH Tensile strength or tenacity is the stress at the breaking point of the test specimen. Tensile strength is obtained from the experimental data using equation $\text{Tensile strength} = \text{Load at break} / \text{Original cross-sectional area} = L / b \times D$ Where L = the load applied in N, b = the width in mm and D = the thickness in mm $\text{Tensile Modulus} = \text{Tensile stress} / \text{Tensile strain} = \text{Difference in load (N)} / \text{Difference in extension (mm)}$

D. IMPACT STRENGTH :

The unnotched Izod impact strength of each sample was tested as per ASTM D 256-88. All the samples were tested as unnotched so that they would be more sensitive to the transition between ductility and brittleness. Specimens having thickness 3.2 mm with 10mm crosssection and 64 mm long were clamped in the base of the pendulum testing machine so that they were cantilevered upward. The pendulum was released and the force consumed in breaking the sample was calculated.

E. FLEXURAL PROPERTIES:

The flexural strength represents the highest stress experienced within the material at its moment of rupture. It is measured in terms of stress. Flexural properties were studied as per ASTM-D 290 using Instron testing machine (Model 6025 UK), at 10 mm/minute cross-head speed, using specimen with a width of 25 mm, length of 200mm and thickness of 3mm. A distance of 115mm is kept in between the grips. Five specimens were tested for each sample.

F. HARDNESS :

Hardness of the composite material was measured using duro meter as per ASTM D2240 specimen with 3mm thickness. The specimen was placed on a hard horizontal surface. The duro meter was held in a position in the point of indenter at least 12 mm from any edge of the specimen. The duro meter had a pointed indenter projecting below the base of the pressure foot. The indenter was pressed into the plastic specimen, so that the

base rests on the plastic materials surface and the amount of Indentation was registered directly on the dial indicator. Hardness was determined at five different positions on the specimen at least 6mm.

VI. Results and Discussion

A. TENSILE STRENGTH

The tensile test results depict that ultimate tensile strength of neat polypropylene is 24.5 MPa. The rose petals that of treated rose petals powder reinforced polypropylene composite is 20.5 MPa. Sample (Wt %) Tensile Strength (MPa) Neat PP 22.5 URPFPP 5 23.3 URPFPP 7.5 24.0 URPFPP 10 22.8 TRPFPP 10 21.5

| Sample (Wt %) | Tensile Strength(MPa) |
|---------------|-----------------------|
| Neat PP | 22.5 |
| URPFPP 5 | 23.3 |
| URPFPP 7.5 | 24.0 |
| URPFPP 10 | 22.8 |
| TRPFPP 10 | 21.5 |

Table 2: Tensile Strength of PP and RPPP composites

Figure 11 shows the ultimate tensile strength (UTS) of neat polypropylene and rose petals powder reinforced polypropylene composites. The tensile strength of chrysanthemum powder reinforced polypropylene composites shows tremendous increase for 5wt% loading and decreases gradually for 7.5wt% and 10wt% loading compared to that of neat homo polypropylene. The polypropylene matrix transmits and distributes the applied stress through the coupling agent (MAPP) to the chrysanthemum reinforcing filler resulting in higher strength. Therefore, the composite can sustain higher load before failure compared to the neat polypropylene. But at higher loading decrease in tensile strength may be due to insufficient coupling; however the strength of 7.5 wt% shows almost equivalent strength as that of neat matrix material.

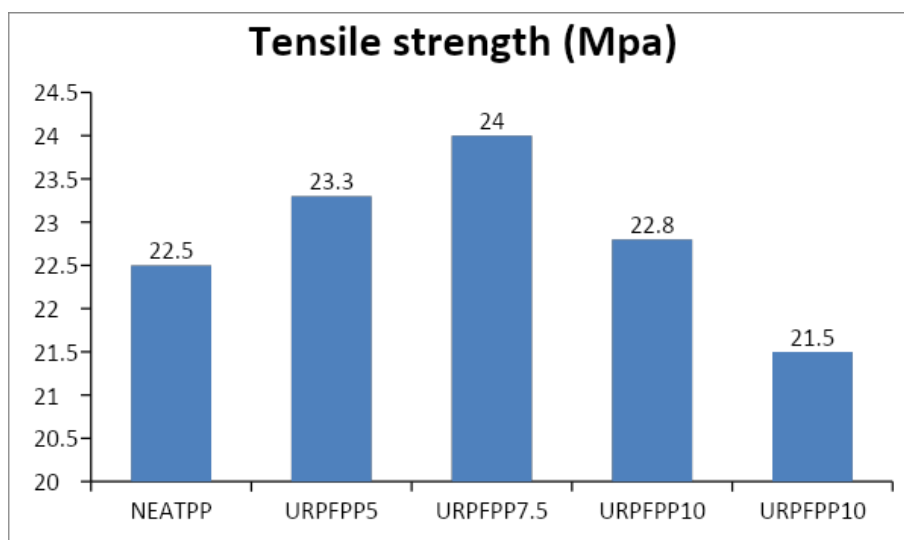


Figure 11: Tensile strength of Neat PP, UCFPP and TCFPP Composites

B. IMPACT STRENGTH :

The impact test results show that impact strength of neat polypropylene is 4.12 KJ/m² . The impact strength of Rose petals filler reinforced polypropylene composites is in the range of 2.9-3.66 KJ/m², and that of treated Rose petals powder reinforced polypropylene composite is 2.35 KJ/m² . Sample (Wt %) Impact Strength(KJ/m²) Neat PP 4.12 URPPP 5 2.90 UCFPP 7.5 2.92 URPPP 10 3.66 TCFPP 10 2.35

| Sample (Wt %) | Impact Strength(KJ/m ²) |
|---------------|-------------------------------------|
| Neat PP | 3.8 |
| URPFPP 5 | 2.1 |
| URPFPP 7.5 | 2.5 |
| URPFPP 10 | 2.8 |
| TRPFPP 10 | 2.2 |

Table 3: Impact Strength of PP and CFPP composites

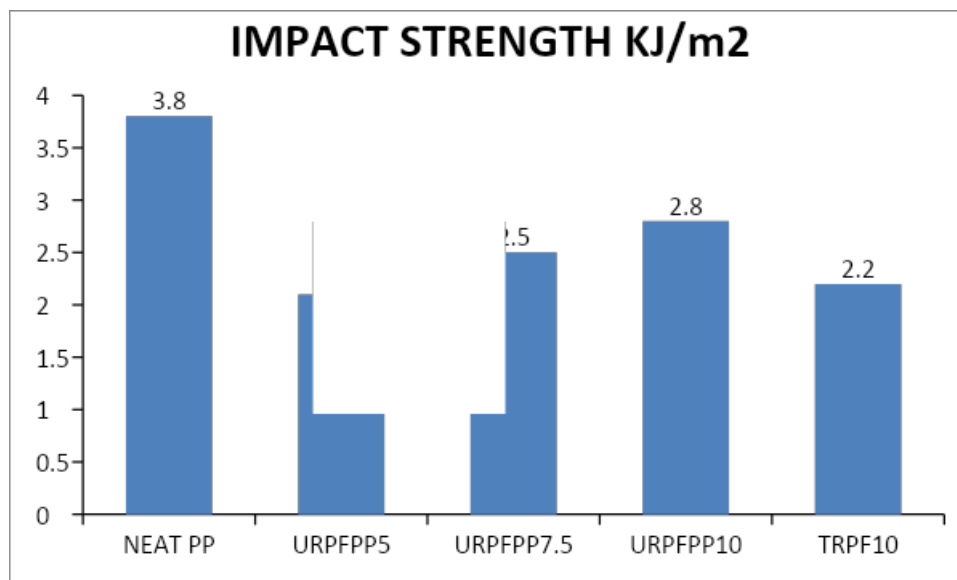


Figure 12: Impact strength of Neat PP, UCFPP and TCFPP Composites

Table 3 shows the impact strength values for neat, coupled and treated rose petal powder reinforced polypropylene composite from which the following conclusions could be drawn. The result of impact test shows an improved the impact strength of 3.8KJ/m² properties for maximum filler content of 10wt% compared to other lower weight percentage composites, thereby increasing the toughness properties of the composite material. This may be probably due to the addition of filler material might have filled the small voids and the regions of particle corners thereby improving the impact strength. In case of treated rose petal reinforced composites, the impact strength was found to decrease comparatively from polypropylene; this may be due to the reason that chemical treatment had decreased the surface polarity thereby increasing the surface roughness. Therefore, response of treated rose petals reinforcement towards impact strength of composites reflects a failure process involving crack initiation and growth in the resin matrix due to fiber breakage, pull out delaminating and disbanding due to chemical treatment.

FLEXURAL STRENGTH :

| Sample (Wt %) | Flexural Strength(MPa) |
|---------------|------------------------|
| Neat PP | 30.5 |
| URPFPP 5 | 34.5 |
| URPFPP 7.5 | 33.4 |
| URPFPP 10 | 31.3 |
| TRPFPP 10 | 40.1 |

Table 4

The impact test results show the impact strength of neat polypropylene is 30.5 MPa. The flexural strength of untreated rose petal filler reinforced polypropylene composites is in the range of 30.5-34.5 MPa, and that of treated chrysanthemum powder reinforced polypropylene composite is 40.1 MPa.

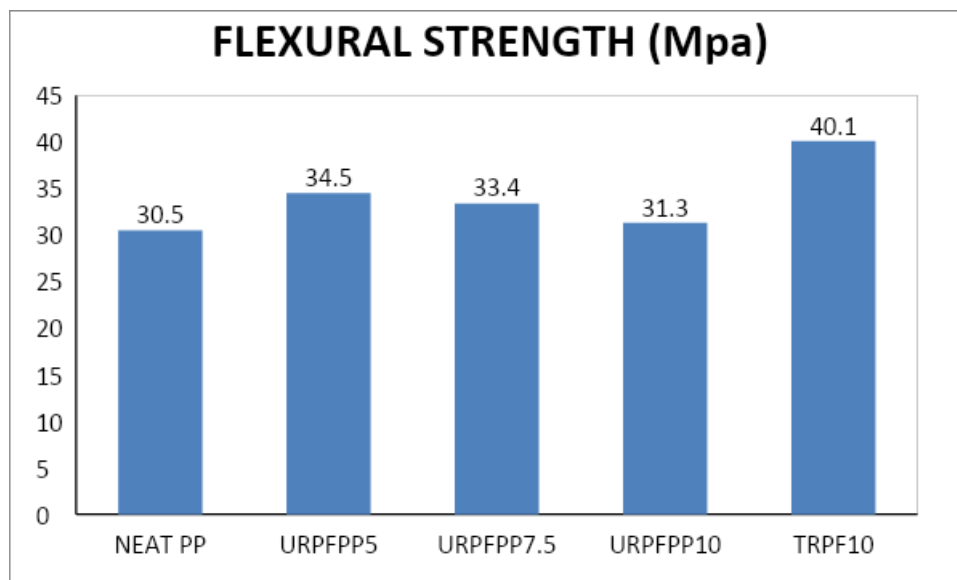


Figure 13

The effect of maleic anhydride on the flexural strength of rose petal composite is shown in Figure 13. As can be seen, flexural strength of composites increases with filler percentage of 5wt% but decreases for 10wt% compared to the controlled matrix material. Whereas treated composite material shows enhanced flexural property. This may be because of the effect of coupling agent enhances the interface adhesion between matrix and that of rose petal filler resulting in higher flexural modulus. But at higher loading shows decrease in flexural strength may be due to insufficient coupling, however the flexural strength of treated composite material show very good improvement when compared to that of neat PP matrix, due to the induced polar group within the matrix through chemical treatment.

C. HARDNESS :

It is well known that hardness implies a resistance to indentation, permanent or temporary deformation of the material. Table 5 shows the hardness of neat polypropylene is 68.8. The hardness of untreated chrysanthe filler reinforced polypropylene composites is in the range of 78.6- 79.8, and that of treated rose petal powder reinforced polypropylene composite is 79.6. Sample (Wt %) Hardness Neat PP 69.1 URPFP 5 73.6 URPFP 7.5,75 URPFP 10 77.8 TRPFPP 10 73.6

| (Wt %) | Hardness |
|-----------|----------|
| Neat PP | 69.1 |
| URPFP 5 | 73.6 |
| URPFP 7.5 | 75.0 |
| URPFP 10 | 77.8 |
| TRPFPP 10 | 73.6 |

Table 4: Hardness of PP and RPPP composites

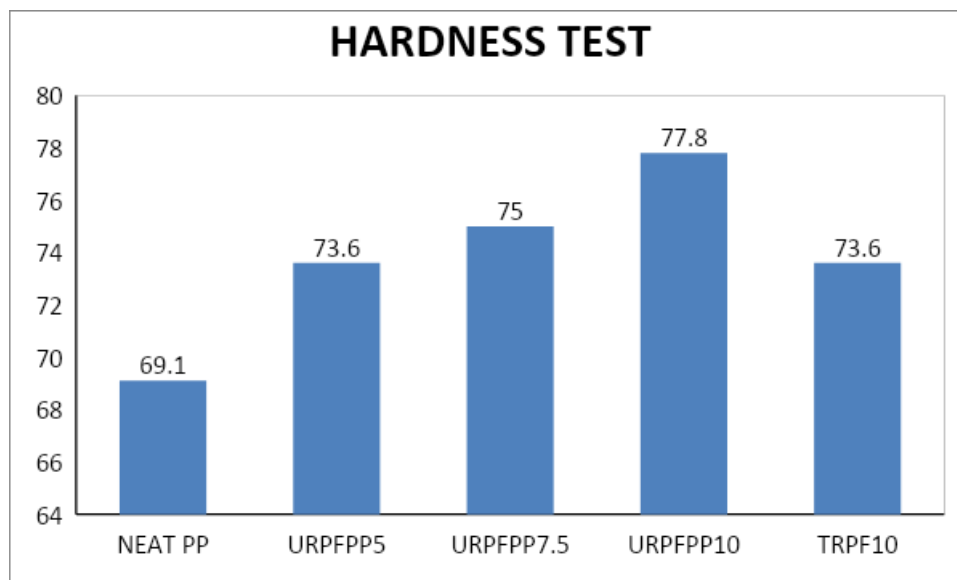


Figure 14: Hardness of Neat PP, URPPP and TRPPP Composites

Figure 14 shows that hardness increases with increase in filler content in case of coupled rose petal powder composites compared to that of controlled matrix material. Maximum hardness is obtained for 10% filler content and that of treated composite material as shown in the figure 13. This reflects the resistance of the chrysanthemum filler (10%) and that of treated filler to deformation in the polypropylene matrix.

VIII. Conclusion

The tensile strength decreases with increase in the filler content. The tensile strength of treated composites shows a higher value than neat polypropylene.

*Impact strength also decreases with filler content, but improved strength is obtained for maximum filler composite material.

* Treated impact material shows a drastic decrease in strength.

*Increase in impact strength of composites with maleic anhydride as a coupling agent will be desirable for making house hold products due to their considerable stability and strength properties.

*The flexural strength of 5wt% is higher than the neat material but gradually decreases with increase in the filler content.

*The flexural strength of treated composites shows a higher value than neat polypropylene matrix.

*Hardness increases with filler content for coupled composite and as well for treated one. Thermal stability increases with increase in chrysanthemum filler content.

*Increase in hardness and thermal stability of maleic anhydride coupled untreated composites will be desirable for furniture parts and making building materials due to their improved hardness .



Figure 15: Moulded product of Rose petals Composite

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