Indian Journal of Pure & Applied Physics
Vol. 59, May 2021, pp. 372-378

Characterization of on Newly Developed Hybrid Reinforced Composite of Polypropylene with Agave –Amaricana, Nano-talc and Starch

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Received 9 May 2020; accepted 5 April 2021

The incrementing demand for green and biodegradable material due to increased awareness and government regulations. This gives a new path to the creation of hybrid composite. This may improve the properties. In the present research work, natural fibres were extracted from Agave Americana plant, starch, and nano-talc reinforced in polypropylene matrix was used to synthesis hybrid composite by twin screw extruder. Maleic anhydride grafted polypropylene was used to enhance the compatibility between natural fibres and starch. Various samples were prepared by varying the content of natural fibres, starch and nano-talc. The prepared hybrid composite tested for its homogeneity by SEM and XRD test. The nano-talc in small content and natural fibres content of 5% gave better properties with enhanced tensile strength, and impact strength. Homogeneous and reactive mixing was reflected in SEM and FTIR, were a good indication for producing cheaper and lighter materials with enhanced mechanical performance.

Keywords: Hybrid composite, Natural fibre, Nano-talc, starch, FTIR, SEM, XRD

1 Introduction

Polypropylene is the most commonly used thermoplastic polymer due to low density, high crystallinity, high stiffness, hardness and tensile strength. The polypropylene is non-degradable which is concern for the environment, and disposal[1]. Hence due to increased awareness and government regulation, there is a demand for environment-friendly material. The biodegradable polymers would future materials. These may be easier to dispose at the finalised stage of the product life cycle[2-3]. Generation of polypropylene required approximately 20 times more energy and higher emissions of exhaust fumes than the creation of natural fibre. Two or more reinforcement in the polymer matrix is used in creating hybrid composites for achieving diversities in the resultant properties. Substitution of matrix polymer by the high fraction of nature friendly fibre and starch would improve the environmental performance, and nano-talc contributes nano size particles properties to the polymeric hybrid composite[4].

The hybrid composite of bio-material (starch), natural fibre (Agave Americano), nano-talc with polypropylene offered a practical approach to this environmental concern with improved mechanical performance. Starch is an important material as filler for composites due to easy availability, as all organic material consists of it, in the form of polysaccharides. The natural fibre is also incorporated as filler to reduce the burden on petroleum resources which are depleting at a very fast rate. Further natural fibres as reinforcement, saves a lot of energy, reduce the emission of CO_2 *i.e.* green and have a good impact on ecology, and more importantly may improve the socio-economical life of rural areas. As they require less energy for production and processing when compared with synthetic fibres[5]. On comparing natural fibres with synthetic fibres it was found that former are easily available, non-toxic, non-corrosive (more machine life), biodegradable, cheaper and have less density. The fillers are hydrophilic. On contracting with environmental conditions the properties got deteriorated[6-9].

Polypropylene, starch, natural fibre did not react chemically to each other. Maleic anhydride grafted polypropylene (MAPP) used as compatibilizer. Natural fibres were alkali treated for better compatibility between polymer matrix and fillers[10-13]. Modification of polypropylene with the addition of maleic anhydride provided polar interaction and covalently links polypropylene to hydroxyl groups of

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the cellulose. This enhanced the properties and adhesion among the fillers of the composites[14-16].

The inclusion of low quantity of nanoparticles to polymer matrix in hybrid composites may lead to improve mechanical performance and water intake capacity. Properly dispersed nanocomposite showed better properties on comparing with the polymer matrix. Talc qualified as good nanoparticle due to its plate type structure with nano metric thickness[17-18]. The degree of agglomeration and crystallization rates was affected by the nanoparticles, amount of nano content and compatibilizer concentration[19]. Nano particles could improve the stiffness, dimensional stability and gas barrier properties of the polymer[20-22]. Nano composites due to presence nano scale particle exhibited improved properties and found the possibility of material design flexibility[23]. Han *et al.*[24] found that applying nano clay on bamboo fibres, filled high density polyethylene composite. Tensile strength, bending modulus and impact strength increased with the content of coupling agent melted anhydride polyethylene (MAPE) whereas the addition of clay led to reduced mechanical properties. Chirholm *et al.* [25] found the effects of micro and nano-sized SiC reinforcement on the mechanical properties of an epoxy matrix system and found that nano particles based composites have superior mechanical properties compared to micro sized composite. Fernandes *et al.*[26] found the effect of coir fibres into a composite of cork and HDPE, and observed that the presence of coupling agent increased in tensile strength and tensile modulus when compared with basic composite. Essabir *et al*.[27] found the effect of the coupling agent (SE BS-g-MA) on tensile strength and observed that tensile strength increased for all hybrid composite when compared to untreated hybrid composites. Mohanty *et al*.[28] investigated the effect of maleic anhydride grafted polypropylene (MAPP) as a coupling agent for surface modification of jute fibre/polypropylene composite. Gilberto Garcia *et al.*[29] evaluated the mechanical properties of epoxy composite reinforced with curaua fibres and organophilic clay, treated with NaOH solution at three levels(2.5,5,10%) and nano clay also at three levels (2.5,5,10wt%) and observed that tensile strength, flexural strength and impact resistance are better for trated fibres. The highest tensile and flexural strengths are achieved with 5wt% of nano clay, 20 wt% of curaua fibres when treated with 5% NaOH for 4 hours.

Su *et al.* [30] investigated the effect of the fillers on the polypropylene/low density polyethylene composites. These fillers drastically improved the tensile strength, strain at break and modulus simultaneously. Arrakhiz *et al*.[31] analysed that tensile strength of alfa fibres/polypropylene reduced by a great extent, but at the same time reported extensive enhancement in young modulus values. Michal *et al*.[32] observed that, scratch resistance increase with the content of talc, and degree of crystallinity of surface layer decrease of polypropylene and talc composites. Uchechi C Mark *et al.*[33] studied the effect of carbonized coconut shell particles from (0-40 wt%) of various sizes and concluded that the reinforced polypropylene composites have enhanced mechanical properties like yield strength, tensile strength, tensile modulus, flexural strength and hardness in proportion to increased amount of filler, however the elongation at break and modulus of resilence decreased with increase of filler loading. Elkhaoulani *et al.*[34] studied the hemp fibres and polypropylene composites and found that young's modulus increased.

The review of papers showed that hybrid composite based on Agave-americana natural fibres and polymer filled with bio-material like starch drew restricted attention for research work. The hybrid composites having biomaterial and natural fibres as reinforcement attracted many researcher and scientists due to advantages like light weight, economical and sustainability. Moreover, the above mentioned hybrid composites finds application in advance sectors like automotive, construction and aerospace. Further trials and work to make and finds its usefulness in additives manufacturing.

An attempt was made through this paper to analyse the change in properties and structure in developing hybrid composite. The ingredients were eco friendly like potato starch, Agave-americana natural fibres, Nano-talc, and polypropylene. The amount of compatibilizer MAPP and starch maintained at 10 and 20% respectively and 1, 3, and 5% agave-americana fibres treated with alkali and nano-talc. The hybrid composites reactive melt was blended in a co-rotating twin screw extruder. The melt extruded samples were evaluated by FTIR to observe the result of melt extrusion and tested for mechanical strength. The fractured surfaces of the composites were investigated by SEM and XRD for the morphology of the hybrid composite.

2 Materials and methods

2.1 Raw material and Hybrid Composite Development

Polypropylene (H350FG) in powder form was purchased from vendor companies. This was used for developing hybrid composites as a matrix. It revealed from the technical data sheet that Melt flow rate of this material was 35 g/10 min at 230 °C /2.16Kg (ASTM D 1238). Potato starch having molecular weight 162.14g was purchased from *Loba Chemie*, India. The maleic anhydride grafted PP (MAPP-EpoleneG-3015)was supplied by Eastman Chemical Products Co (USA).The agave-amricana natural fibres were extracted from Agave Americana plant by water retting process. Nano-talc, $3MgO.4SiO_2.H_2O$ (EI-GT0.7WG) with particle size under 100 nm was purchased from a vendor company.

The reactive melt blending process was used to produce the hybrid composite as this process was more effective, economical and environmentally friendly as no organic solvent needed for mixing ingredients. This process could be used for industrial mass production[35-36]. Natural fibre extracted from Agave Americana plant by retting process, which was available on our university campus. Alkali treatment of natural fibres increased surface roughness formed hydrogen bonds. This enhanced better bonding between the matrix and reinforcement[37]. The starch was mixed with PP in nano-talc, natural fibre with experimental formation as shown in Table 1. All the constituents were mixed in a high speed mixer for two minutes and melt extrusion technique was used for composite formation. The temperature profile was from 170-190 °C for the twin screw extruder, screw rpm was 275, Melt Pressure was 25 MPa during extrusion. The melt extruded composite was cooled in a water bath and then pelletized into small granules of size 2-3 mm.

2.2 Test specimen preparation by Injection Moulding

The granules of hybrid composites obtained by melt extrusion were injection moulded into standard mechanical test specimen for tensile strength and flexural strength as defined in ASTM standards and plaque (square plate) of 0.5mm thickness and 2x2 inch were moulded on an injection moulding machine. The Injection Moulding process parameters were given in Table 2. The injection temperature kept lower than the degradation temperature of a hybrid composite as 190 °C to retain properties of composite, because at higher temperature natural fibre and starch may degrade. This may affect the test values.

2.3 Methods for testing properties

Elongation in the material was tested by the tensile testing machine. The samples were fabricated in dumbbell shape. The test was performed until breaking point. Flexibility was checked by using three-point bending mode. The value of force was recorded at the breaking point. The material at rest position may bear a higher load. But, it may fail under a small amount of load with impact. The material was tested for impact load at 2.75 J. Each sample was tested ten times for calculating the final value for every respective category of strength.

3 Results and Discussion

3.1 Mechanical Properties:

The properties were improved. This may be due to the improved interfacial bonding in the presence of compatibilizer and plasticization of starch. The sample no. 2 was more suitable composite having the tensile strength equal to pure polypropylene but its lost ductility as the elongation at break abruptly reduced. This may be attributed to that polypropylene being filled with more rigid fillers. This confirmed that the addition of fillers especially starch and fibres restricted the motion of polymer chains and hence

Table 1 — Formation of hybrid composites based on polypropylene

S.No.	Sample	Polpropylene (PP)%	Starch (ST)%	Nano-talc (TL)%	Natural Fibre (NF)%
1	PP	100	00	00	00
2	PPST20TL01NF05	100	20	01	05
3	PPST20TL03NF03	100	20	02	03
4	PPST20TL05NF01	100	20	05	01
5	PPST00TL05NF00	100	00	05	00

Table 2 — Injection moulding process parameter for moulding of test ting specimen

Temperature (Z1) 155°C	Temperature (Z2) 153°C	Temperature (Z3) 150°C	Temperature (Z4) 150°C
Melt Temperature 165°C	Screw Speed (rpm) 55	Injection Speed (rpm) 45	Injection Time (Sec.) 8
Mould Cooling (Sec.) 15	Hold Time (Sec.) 2	Cycle Time (Sec.) 35	

Table 3 — Tensile strength and elongation at break for PP& Hybrid composites

S. No.	Sample	Tensile Strength (MPa)	Elongation Break %
1	PP	32.792	277.433
2	PPST20TL01NF05	31.035	13.100
3	PPST20TL03NF03	27.963	9.133
4	PPST20TL05NF01	29.362	11.500
5	PPST00TL05NF00	29.616	13.83

elongation decreased sharply. This sample verified that nanomaterials presence was more effective at low content after it fillers started to form agglomerates due to high surface energy of nanomaterials. The results obtained showed that an optimum level of talc and natural fibres gave a better mechanical performance. Tensile strength was sensitive to the degree of dispersion level. The results are shown in Table 3 and Fig. 1(a-b).

3.2 Flexural Strength

The flexural strength for sample no. 2 was slightly improved as compared to the pure polypropylene. This sample consists of a higher level of fibres loading and fibres contributed against bending forces, and thus flexural strength improved. A high amount of talc had the property to agglomerate and thus produces negative effect on the mechanical properties. These results are given in Fig. 2 and Table 4.

3.3 Impact Strength

The impact strength of hybrid composites increased slightly in sample no. 2 then it increased sharply in sample no. 3 & 4. The crack was generated due to an impact propagated towards the poor interfacial sites, while in the case of sample 3 and 4 the presence of *agave-americana* fibres bore the load and improved impact strength about 15%. The results are shown in Fig. 3 and Table 5.

3.4 Analysis of SEM

The SEM micrographs of PP and its hybrid composite are given in Fig. 4. These images with different percentage of filler by weight revealed that fillers are being continuously and uniformly mixed throughout the polypropylene matrix and form a homogeneous phase. Results showed that fillers were properly dispersed due to the use of compatibilizer and extrusion conditions. The maleic anhydride facilitates the strong ester bonds between the PP chains and OH group on the fibres which were identified in FTIR test.

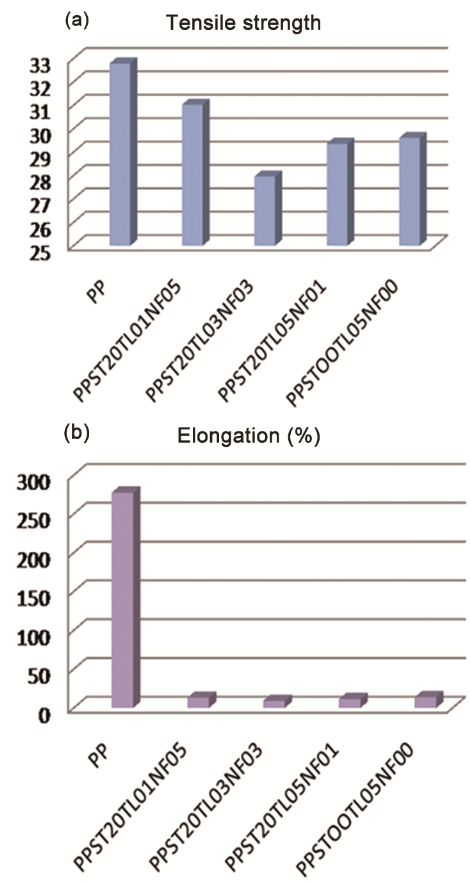

Fig. 1 — (a) Tensile strength, and (b) Elongation at break (%) for PP and its hybrid

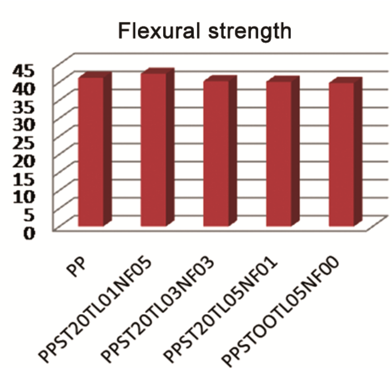

Fig. 2 — Flexural strength (MPa) of PP and its hybrid composites

Table 4 — Flexural strength of polypropylene based hybrid composites

S. No.	Sample	Flexural Strength (MPa)
1	PP	41.29
2	PPST20TL01NF05	42.359
3	PPST20TL03NF03	40.275
4	PPST20TL05NF01	40.198
5	PPST00TL05NF00	39.765

Table 5 — Impact strength of polypropylene based composites

S. No.	Sample	Impact Strength (kJ/m^2)
1	PP	1.80
2	PPST20TL01NF05	1.81
3	PPST20TL03NF03	2.01
4	PPST20TL05NF01	2.04
5	PPST00TL05NF00	1.88

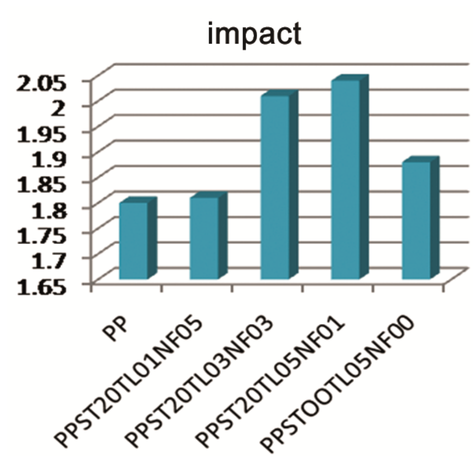

Fig. 3 — Impact strength (kJ/m^2) of PP and its hybrid composites

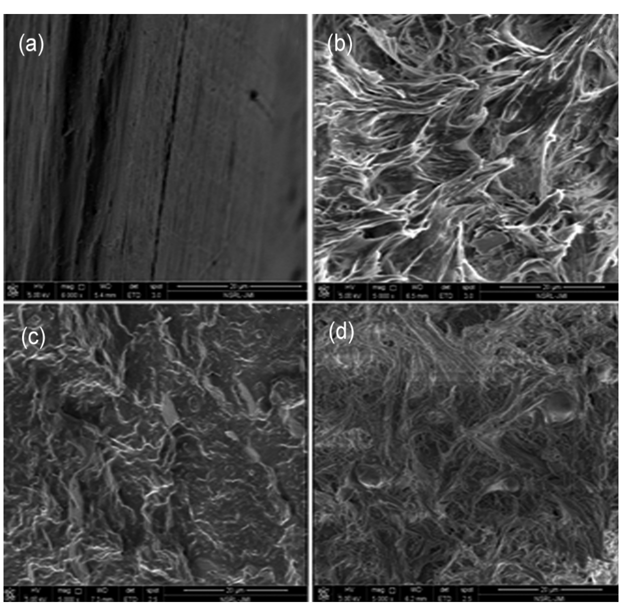

Fig. 4 — SEM images of PP and its hybrid

3.5 Analysis of X-ray diffraction

The XRD pattern of PP and its hybrid composites is given in Fig. 5. The sample no. 1 (Pure PP) shows the sharp peaks at 2θ values of 14.3, 17, 18.7 and 22 for the corresponding values to the (110), (040), (130), and (041) planes respectively for pure Polypropylene system. These crystalline peaks were presented in other hybrid composites *i.e.* sample no. 2 to 5, confirmed the presence of PP as polymer matrix in all composites. Sample no. 2, 3 and 4 showed extra peaks at 25.5, which confirmed the presence of starch. The existence of talc in the hybrid composites sample no. 2,3,4 and 5 could easily be characterized by the diffraction peak at 2θ values 9.3° and 28.62° corresponding to the (002) and (113) plane respectively.

3.6 Fourier Transform Infra Red Spectroscopy (FTIR)

This spectrum peaks were utilized to confirm the existence of reactive melt blending and result with the bond formation between polypropylene and its fillers *i.e.* starch, agave-americana and nano-talc through compatibilizer MAPP, which ultimately leads to the developments of hybrid composites. All five spectrums shows peaks of polypropylene at 2951, 2868 and 1376 cm^{-1} ($-CH_3$ groups), 2917, 2837 and 1457 cm^{-1} ($-CH_2$ groups), and three characteristic isotatic peaks at 1167, 997, and 973 cm^{-1}. This confirmed the presence of polypropylene in all composites as matrix. Peaks at 1746 cm^{-1} confirms anhydride group of MAPP in the backbone of polypropylene, through which all fillers becomes the part of structure and contributes their properties in hybrid composites[10]. The existence of starch in

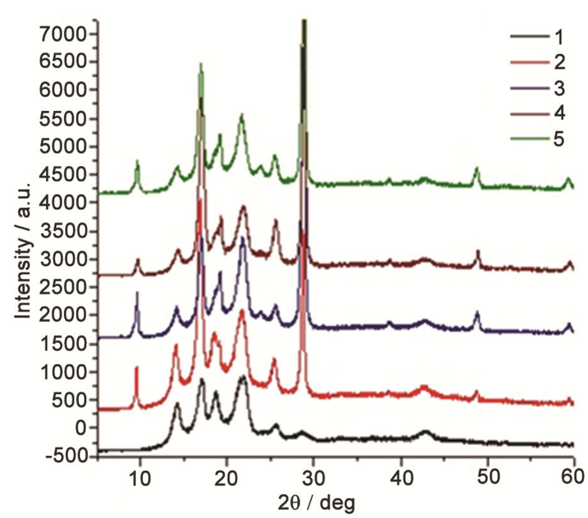

Fig. 5 — X-ray Diffracto graph of PP and its hybrid composites

formulation 2,3, and 4 confirms by very dim humplike the structure at 3600 to 3100cm^{-1} (OH), 1746 cm^{-1} (C=O), 1257 cm^{-1} (-O-C=O) and 1017 cm^{-1} (C-O)[10,43]. The sharp peaks observed at 668 cm^{-1} stretching vibrations of -OH confirms the presence of talc materials and same has been evident from the intensity level of the peaks increases as loading of talc increases in sample no. 2, 3, 4 and 5 respectively.

4 Conclusions

The hybrid composite based on Polypropylene reinforced by starch, *agave-americana* natural fibres and nano-talc were prepared and investigated. Four sets of composites prepared by reactive melt blending using MAPP as coupling agent and its concentration was kept constant at 10% in all compositions while 20% starch in sample no. 2, 3 and 4. The concentration of *agave-americana* and nano-talc was varied from 1, 3 and 5%. The mechanical and morphological properties of composites were studied.

1. These results confirm and provide a scope of new development of a hybrid composites, which require less energy during processing, reduced CO_2 emissions, easily degradable and easy to dispose of due to presence of bio-materials (Starch and *agave-americana*).
2. These materials may reduce the burden on the petroleum products, Natural fibres /starch are easily available and renewable and hence, green/sustainable and environmental/eco-friendly materials.
3. The mechanical characterization showed that flexural strength almost remains constant throughout all the samples. The impact strength increased in sample no. 3 and 4, this reflected that incorporation of *agave-americana* fibres increased impact strength as expected.
4. The tensile strength and % elongation decreased by the addition of a*gave americana*, starch and nano-talc in sample no. 2, 3, 4 and 5. It has been observed that starch and talc participated in hybrid composition as particulate materials as reflected by sharp and crystalline peaks in FTIR, SEMand XRD.
5. The morphological characterization based on scanning electron microscopy (SEM) has confirmed that natural fibres were distributed uniformly indicating a good dispersion of natural fibres, starch and nano-talc in the PP/MMPP.

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