Agave nonwovens in polypropylene composites – Mechanical and thermal studies

***Maya Jacob John*[1, 2] *, Nokuzola Sikampula*[1] *and Lydia Boguslavsky*[1]**

[1]*CSIR Materials Science and Manufacturing, Fibres and Textiles Competence Area,*
P.O. Box 1124, Port Elizabeth 6000, South Africa
[2] *Department of Textile Science, Faculty of Science, Nelson Mandela Metropolitan*
University, P.O. Box 1600, Port Elizabeth 6000, South Africa,

Abstract

Blends of agave fibres with wool waste, pineapple leaf fibres and polypropylene fibres were manufactured by needle-punching technique. Composites were prepared with polypropylene matrix by the process of compression moulding. The effects of blend nonwovens on the mechanical and dynamic mechanical properties of composites were investigated. Composites containing agave-polypropylene (A-PP) nonwovens exhibited superior mechanical properties compared to the other two. Storage modulus of the composites was found to be maximum for agave-pineapple (A-PALF) composites due to the increased stiffness of the composites. Damping was found to be decreased with incorporation of agave nonwovens. As part of product development, parcel trays for automotives were developed from A-PP nonwovens by compression moulding technique. This study looks at the effective utilization of plant and animal fibre waste in composites.

Key words: Agave fibre, wool waste, mechanical properties, DMA,TGA, storage modulus, compression molding

Corresponding author
E-mail: mjohn@csir.co.za, mayajacobkunnel@yahoo.com
Telephone number: ++ 27 41 508 3292

1. INTRODUCTION

The use of green materials (natural fibres and biocomposites) in advanced industrial applications is on the increase. In global terms, South Africa (SA) ranks 6th in terms of the total number of vascular plant species. The importance of South African flora is further realised considering that South Africa occupies only 2% of the world's surface area, but is home to nearly 10% of the world's plant species. An abundant presence of plant species and fibres means that South Africa can significantly contribute to the emergence of this green revolution.

Agave fibres are obtained from the leaves of the plant *Agave Americana (AA)* which are found in abundance in the arid highland areas of SA. The best known and most common application of the Agave plant is the production of alcoholic beverages, such as tequila, by fermentation of the heart (pina) of the plant. However, the bulk of the plant (including the leaves) are not utilised and are considered as waste. A multi-disciplinary study on local agave plants was launched by the Council of Scientific and Industrial Research (CSIR) in SA to evaluate the potential of the agave leaves with a view to adding maximum value to the current production of alcoholic beverages.

SA is also one of the major wool producing countries in the world. In the textile industry, the accumulation of waste wool fibres and their products, which are difficult to decompose in natural conditions because of their high content of cystine residues, has caused important ecological and sanitary problems all over the world [1,2]. Since about 50 wt.% of these waste wool fibres are made of keratin, a usable protein, they have been considered as an attractive renewable material due to their excellent mechanical and thermal properties [3]. Keratin extracted from waste wool fibres has been successfully employed to create biocompatible films and bio-scaffold for medical use [4]. A recent study investigated the use of wool fibres in polyester and found that chemical treatment of wool fibres resulted in improved properties [5]. Thus, the use of waste agave and wool fibres as reinforcement in polymers gives good opportunities for the effective utilization of such wastes.

Recently, researchers have explored the use of agave fibres in various matrices. Mylsamy and Rajendran [6] investigated the mechanical properties of untreated and alkali treated agave fibre reinforced epoxy composites. They observed increased interfacial adhesion in the treated composites resulting in enhanced mechanical properties. The authors also looked into the wear behavior of the composites and observed that at shorter fibre lengths, the wear resistance of the composites increased

[7]. In another interesting study, the adsorption capacity of agave fibre reinforced HDPE composites coated with chitosan was evaluated. The authors [8] used atomic absorption spectroscopy (AAS) to measure metal uptake and claimed that it is a low cost recycled material and can be used to adsorb metal ions from polluted waters. The use of pineapple leaf fibres and its improved mechanical performance have been explored in geopolymer polymer composites [9]. The properties of agave fibres in polystyrene was investigated by Singha and Rana who reported on the improved mechanical properties of the composites [10].

The use of agave fibres in waste plastic was extensively studied by Thamae et al [11]. The authors found mercerization of agave fibres resulted in improved properties. The effect of different chemical treatments on agave fibres in polyester composites was investigated by Bessadok et al [12]. The different treatments evaluated were acetic anhydride, styrene, acrylic acid and maleic anhydride. The main observation was that the chemical treatments reduced the overall water uptake of agave fibres. Similar results were observed by Reddy et al. who observed an increase of crystallinity in alkali treated agave fibres resulting in improved tensile properties [13].

The properties of blue agave fibres were investigated by Satyanarayana et al. [14]. The authors observed that the fibres exhibited 70% crystallinity and that tensile properties of fibres were comparable to other lignocellulosic fibres. Blends of agave fibres with polystyrene were prepared by Moscoso et al. [15] and the mechanical and morphological properties were evaluated. They observed that that tensile strength and water uptake increased with increasing fibre content.

This paper investigates the mechanical and viscoelastic properties of agave fibres in polypropylene composites. In this paper, we have used three types of agave fibre blends namely agave–pineapple fibre (A-PALF), agave-wool waste fibre (A-WW) and agave-polypropylene fibre (A-PP) for the preparation of composites. Techniques like thermogravimetric analysis and dynamic mechanical analysis were used to characterize the composites.

2.EXPERIMENTAL

2.1 Materials

Agave fibres (extracted from agave leaves) and pineapple fibres were obtained from local sources in Graaf-Reinet and Bathurst respectively. Wool waste was collected from Dorper sheep – a variety of sheep grown exclusively for meat - from KGB, Port Elizabeth. Polypropylene in sheet form (6 mm thickness), with a density of 0.9g/cc and melt flow index of 1.5g /10 min was procured from Ampaglas SA.

2.2 Extraction of agave fibres and preparation of nonwovens

Leaf samples were collected from Agave Americana plants cultivated at local farms for fibre extraction and investigation of fibre properties. All leaves were processed on a sisal decorticator modified for the extraction of agave fibres. Fibres were extracted from the fresh leaves, then rinsed and dried in open air. Dried fibres were then processed on the opening machine. To make fibres suitable for further processing into nonwovens, fibres were mechanically cottonised on Temafa cottonization line that resulted in reduction of fibre length and fibre diameter (fibre linear density). In this study, the cottonised agave fibres were blended with cottonised pineapple leaf fibres (PALF), wool waste (WW) and polypropylene (PP) fibres in the ratio 90/10 respectively and processed into needle-punched nonwovens. The carrier fibres (PALF, WW and PP) were added to improve the cohesion between fibres. The needle-punched nonwovens exhibited an areal weight of ± 300 g/m2 and thickness of 1.2 mm. Figure 1 a, b and c presents the pictures of A-PALF, A-PP and A-WW nonwovens respectively.

(a)

(b)

(c)

Figure 1: Pictures of (a) A-PALF, (b) A-PP and A-WW nonwovens

2.3 Preparation of composites

Composites were prepared from agave blend nonwovens and polypropylene. The agave blend nonwovens were cut into small uniform squares (30 cm x 30 cm) and were dried in an air oven at the temperature of 110°C for 7 h. The dried nonwoven mats were randomly arranged and placed between weighed polypropylene sheets. This was wrapped in Teflon® sheets and sandwiched between two aluminium plates. These two plates were then placed between the two platens of compression moulding press and cured at a pressure of about 35 bar for 20 minutes at 190°C, followed by cooling under pressure for 3 minutes. The weight fraction of all the composites was maintained at 30%.

3. Analysis

The bundle tensile testing of agave fibres were carried out using an Instron Tensile Tester (Model 3345). The sample preparation was done according to ISO 3060 at a crosshead speed of 30 mm/min. Ten bundles were tested per sample. All samples were conditioned at a temperature of 21±1°C and relative humidity of 65±2 % for at least 24 hours before testing.

Tensile and three-point bending tests of composites were carried out using an Instron Universal Testing Machine, model 3369. Tensile testing (sample dimensions 15 x 10 x 3 mm) was measured according to ASTM methods D638 at a crosshead speed of 50 mm/min and a gage length of 50 mm. Flexural testing (sample dimensions 127 x 12.7 x 3 mm) was carried out in accordance with ASTM D-790, at a crosshead speed of 5mm/min and a span length of 60 mm. 4 replicates were tested for both tensile and flexural tests.

Dynamic mechanical analysis was carried out using the Perkin Elmer DMA 8000. Samples of dimensions 50 x 12 x 3 mm were used for testing. The testing temperature ranged from -20°C to 150°C and the experiment was carried out 1 Hz. The samples were tested under dual cantilever mode at strain amplitude of 0.05mm.

Thermogravimetric (TGA) studies were carried out using a PerkinElmer TGA in an inert atmosphere at a heating rate of 10^{o}C/min. The temperature range used for the analysis is 30 ^{o}C to 700 ^{o}C.

4. Results and Discussion

4.1. Mechanical properties of agave fibres

Table 1: Mechanical properties of fibres

Fibres	Bundle strength tenacity at maximum load [cN/tex]	Textile extension [mm]	Fibre diameter [µm]	Fibre linear density [tex]
Agave	26-11	1.4	140-175	19-30
Flax	31-37	3-7	40-620	0.4-10
Ramie	29-41	3-7	16-904	-
Pineapple	26-44	0.45	20-200	2.5-5.5
Hemp	25-37	2-3	25-500	5.4-40
Sisal	21-35	1.2	9-460	15-20
Wool (single fibre)	10-20	25-60	17-32	10-50

Table 1 presents the tensile strength of the extracted agave fibres and commonly used leaf and bast fibres [16]. The values for the other bast fibres were taken from literature. It can be observed that the tenacity exhibited by agave fibres is lower when compared to bast fibres like flax and hemp but similar to that exhibited by sisal fibres [17]. The strength of natural fibres is largely dependent on the cellulose content and microfibrillar angle. As agave fibres possess lower cellulose content (60%), the fibres exhibit lower tensile strength compared to bast fibres.

4.2. Mechanical properties of composites

Figure 2 presents the tensile strength of the composites. The incorporation of agave nonwovens in virgin polypropylene results in a general increment of tensile properties. Among the composites, it can be observed that tensile strength of composites containing pineapple leaf fibres is the highest. This can be attributed to the fact that pineapple leaf fibres contain 80% cellulose and are one of the strongest fibres. The decrease in tensile strength in agave-PP composites may be attributed to poor interfacial bonding between PP and agave nonwovens.

The modulus which is a measurement of stiffness and an indication of load bearing capacity also increases with fibre reinforcement (Figure 3). Tensile modulus is mainly dependent on fibre volume fraction [18] and fibre type and composites containing a blend of agave and wool fibres exhibit the highest stiffness.

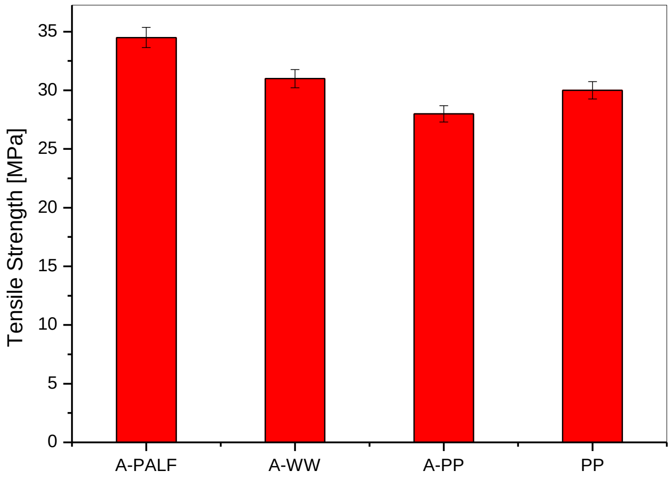


Figure 2: Variation of tensile strength with composites

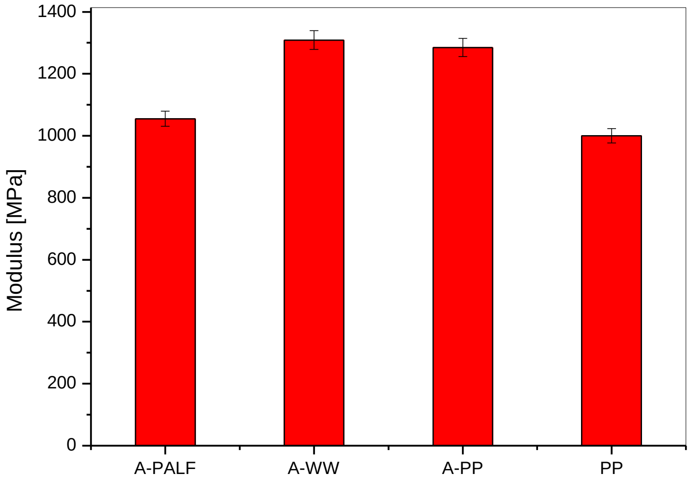
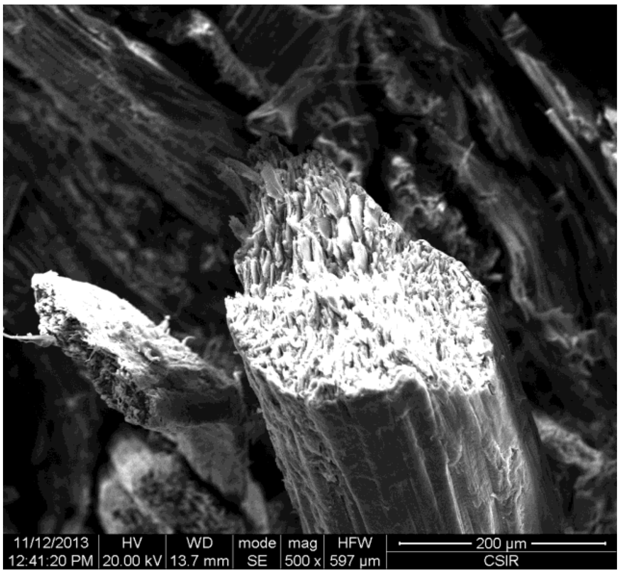
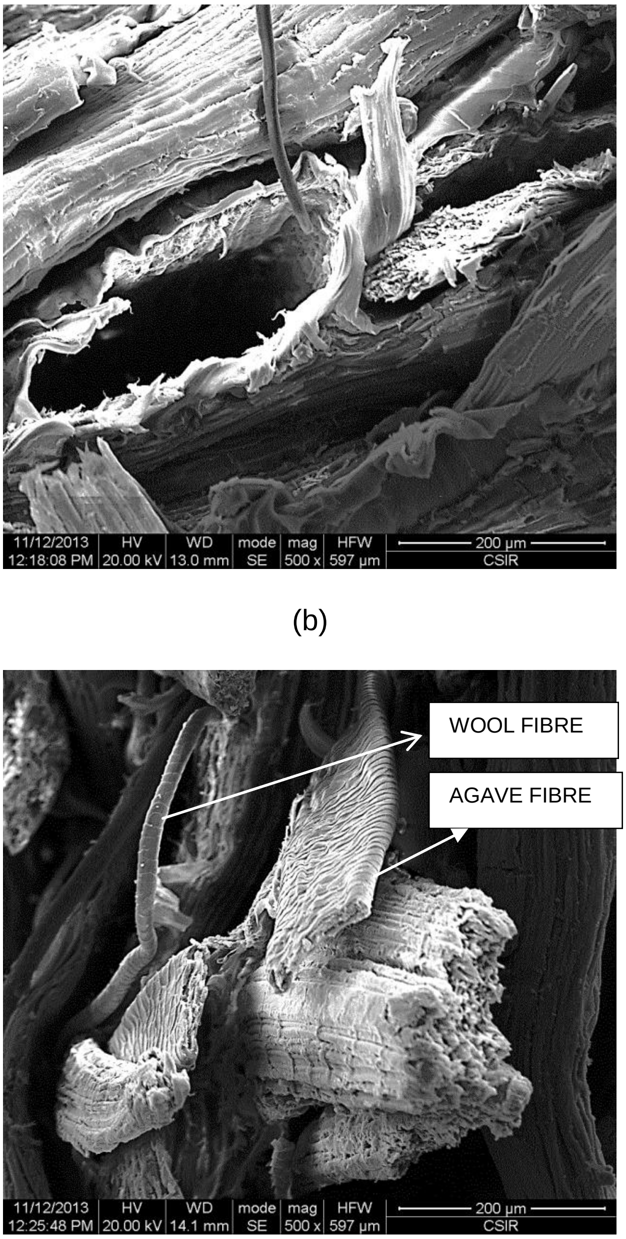


Figure 3: Variation of modulus with composites

SEM micrographs of tensile fracture specimens of A-PALF, A-PP and A-WW are presented in Figure 4 (a-c) respectively. Broken fibre ends can be seen in Figure 3a indicating interfacial interactions. Figure 3b shows the presence of cavities signifying fibre pull-out. This also supports the lower tensile strength seen in A-PP composites. The presence of wool fibres can be clearly seen in Figure 3c along with broken fibre ends of agave fibres. The high modulus and flexural strength can be attributed to the inherent strength of wool fibres and the possibility of wool fibres acting as protein bridges in the matrix.



(a)



(b)

(c)

Figure 4: SEM micrographs of (a) A-PALF (b) A-PP (c) A-WW composites

Flexural strength is a combination of the tensile and compressive strengths, which directly varies with the interlaminar shear strength. In flexural testing, various mechanisms, such as tension, compression and shearing take place simultaneously. It can be observed that the composite containing agave and wool waste exhibits the highest flexural strength (Figure 5). Fan and Yu studied the properties of cortical cells (extracted from waste wool fibres) reinforced chitosan films [19]. They observed an increase in mechanical properties at high loadings and this was attributed to the adhesion between chitosan and cortical cells.

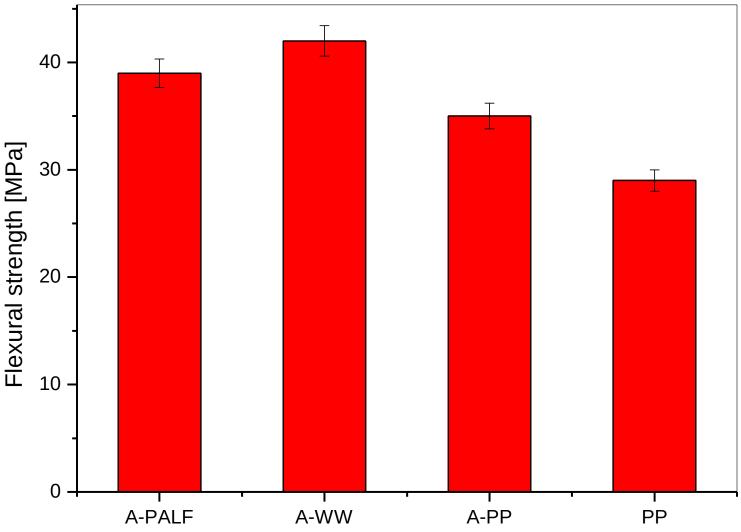


Figure 5: Variation of flexural strength of composites

4.3. Viscoelastic properties

Dynamic mechanical analysis (DMA) can be used to evaluate the performance of composites over a wide range of temperature. Natural fiber reinforced polymers are used to a great extent for the interior lining of cars and commercial vehicles. For these applications, the strength and stiffness properties have to satisfy the requirements of low temperatures of about –20°C up to temperatures of 100°C. Dynamical mechanical analysis is very useful for establishing such a wide range of temperature-dependent material data.

4.3.1 Storage modulus

A clear understanding of the storage modulus –temperature curve obtained during a dynamic mechanical test provides valuable insight into the stiffness of a material as a function of temperature. Figure 6 displays the variation in storage modulus with temperature at a frequency of 1 Hz. It can be observed that the storage modulus decreases with temperature. This reduction in modulus is associated with the softening of the matrix at higher temperature. Among the different composites, agave-PP reinforced polypropylene composites exhibited the highest storage modulus indicating greater stiffness. The addition of fibres allows effective stress transfer at the interface, which consequently increases the storage modulus. The storage moduli of agave-WW and agave-PALF composites were found to be comparable.

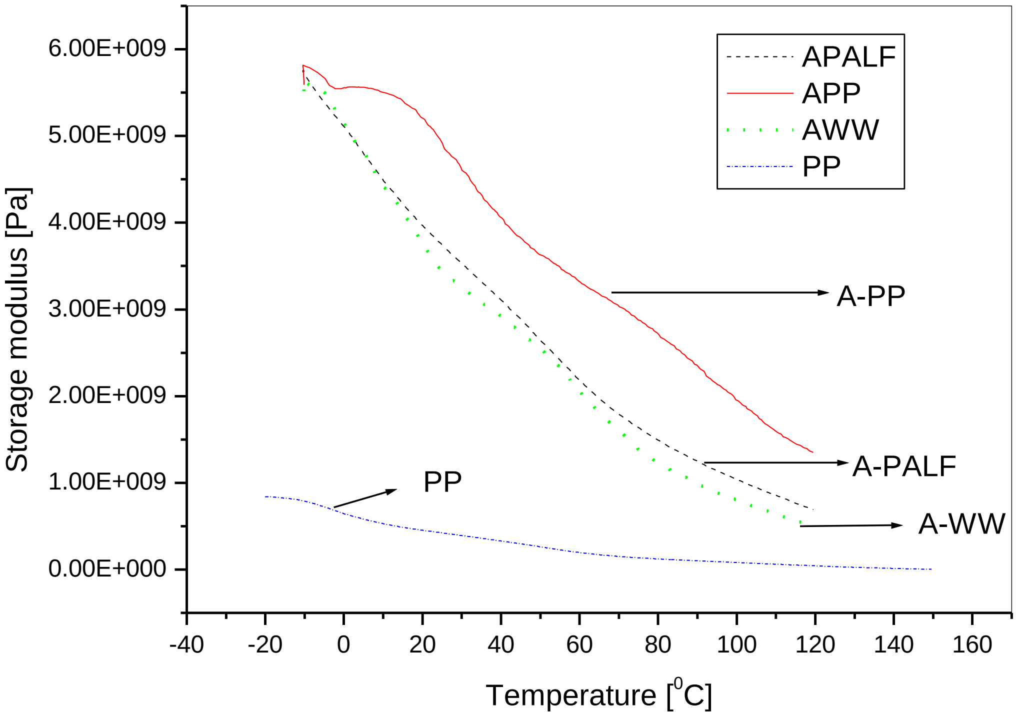


Figure 6: Variation of storage modulus with temperature of composites

4.3.2 Damping

Damping is an important parameter related to the study of dynamic behaviour of fibre reinforced composite material. The major contribution to damping in composite is due to (a) nature of matrix and fibre (b) nature of interphase (c) frictional damping due to slip in the unbound regions between fibre and matrix interface or delaminations and (d) damping due to energy dissipation in the area of matrix cracks and broken fibres [20]. As the damping peak occurs in the region of the glass transition where the material changes from a rigid to a more rubbery state, it is associated with the segmental mobility within the polymer structure all of which are initially frozen in. Therefore higher the tan δ peak value, greater is the degree of molecular mobility. The damping parameter was found to be maximum for polypropylene and with incorporation of nonwovens, the peak of tan δ was seen to decrease (Figure 7). Fibres act as barriers to the mobility of polypropylene chains leading to lower flexibility and lower degrees of molecular motion which results in lower damping characteristics. Amongst the composites, agave-PP blends exhibit the lowest tan δ. This is attributed to better bonding in agave-PP blend composites as the matrix was PP. Wool is a protein fibre and in agave–wool waste composites, the relatively higher damping could be attributed to lack of compatability between wool and polypropylene.

The tan δ curve of PP exhibits two relaxation peaks located at 1.2693 °C (β) and at 68.4810 °C (α). The β transition is related to relaxation of unrestricted amorphous chains of PP and the α-transition is related to the relaxation of restricted crystalline phase of PP and can be attributed to lamellar slip mechanism and molecular chain rotation in crystalline phase. It can also be seen that upon incorporation of agave nonwovens, the position of β-relaxation is not significantly altered.

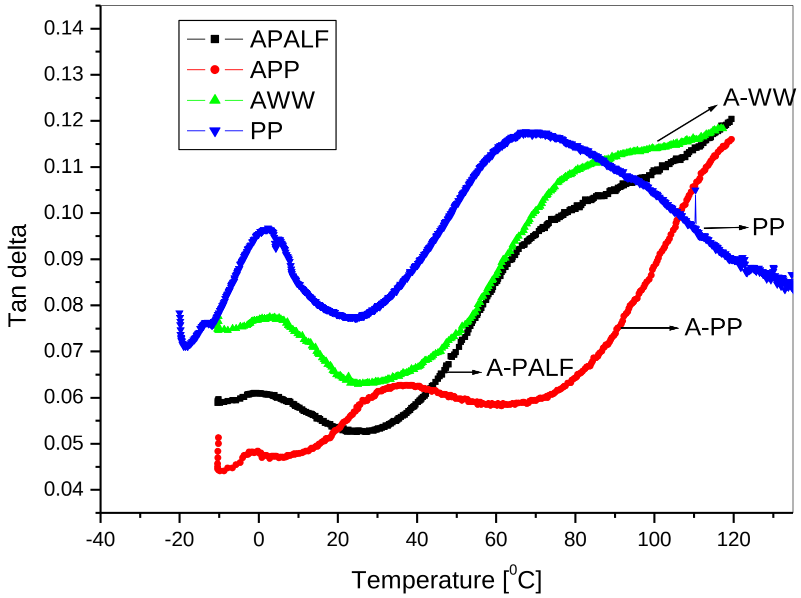


Figure 7: Variation of tan delta with temperature of composites

4.4. Thermogravimetric analysis

The thermal stability in terms of peak degradation temperature and degradation profiles of the different composites are presented in Table 2 and Figure 8 respectively. Lignocellulosic fibres have been observed to degrade between 250 – 500°C; hemicelluloses mainly between 200-350°C, cellulose between 275-400°C and lignin between 250-500°C [21]. As can be seen the degradation of polypropylene is a one step process and the major peak is observed around 501.8°C. In the composites, two peaks were obtained; a minor peak at at 410°C due to hemicellulose and α-cellulose degradation and the major peak around 480°C. The addition of agave nonwovens results in a slight decrease of degradation temperatures. This slight decrease may be attributed to lower lignin content in agave fibres when compared to other natural fibres like kenaf and flax fibres [22]. In weight loss terms, the decomposition temperature at 30% weight loss occurred at 468°C for A-PP, 403°C, 445°C for A-WW and A-PALF respectively (Table 2). Composites containing agave-pineapple fibre blend exhibited a slightly higher thermal stability compared to the composites containing the other fibre blends.

Table 2: Peak degradation temperatures and temperatures at 30% and 90% mass loss

Sample	Peak degradation temperature [^{o}C]	Temp at 30% mass loss [^{o}C]	Temp at 90% mass loss [^{o}C]
PP	501.8	468	496
A-WW	475	403	510
A-PP	475.2	440	496
A-PALF	480.9	445	517

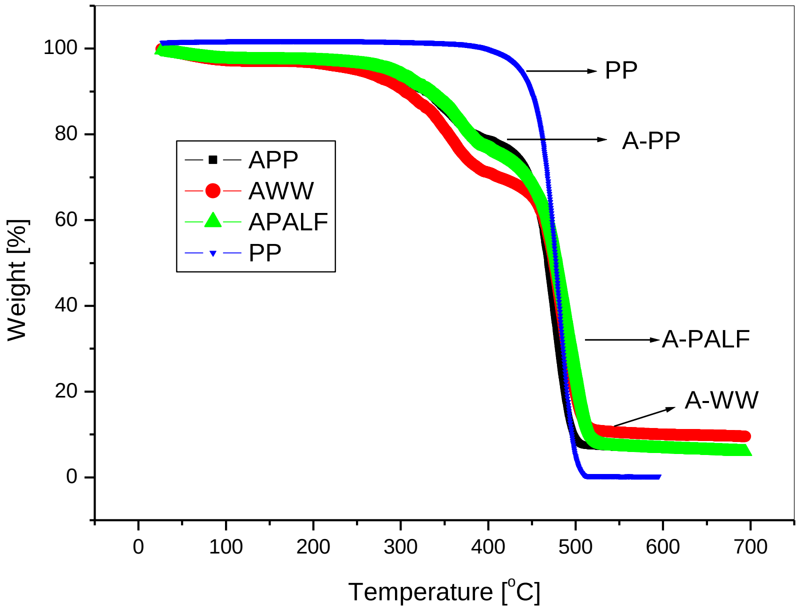
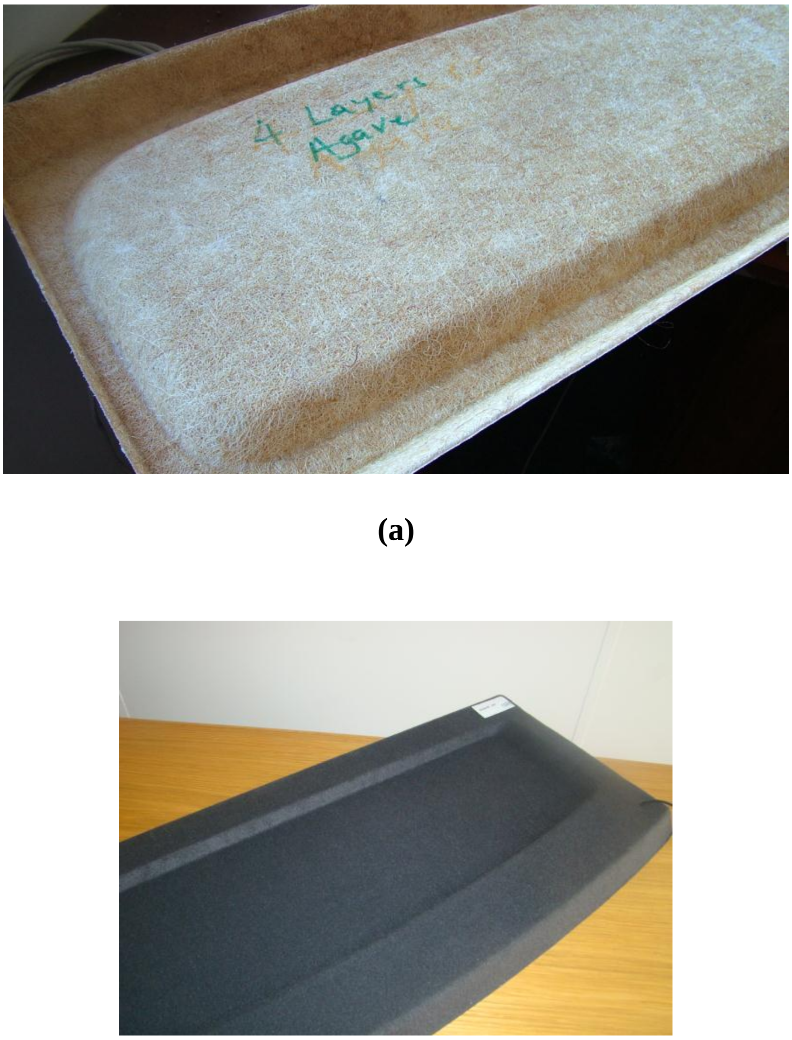


Figure 8: Thermograms of composites

4.5 Product Development

Natural fibre reinforced composites are now being increasingly used in the automotive industry. There is considerable pressure for automotive OEMs to meet 85% Recyclability and 95% Recoverability targets by year 2015. To meet the RRR (Reuse, Recycle & Recover) and the ELV (End of Life) regulatory requirements, increased use of natural fibre based composite/ biopolymers has become unavoidable [23]. The advantages of using natural fibres in automotive applications are that they exhibit comparable specific strength to glass fibre while registering a weight savings of between 10% and 30% and corresponding cost savings. The other appealing factor is that natural fibres are renewable and biodegradable thereby creating a positive environmental impact. Bast fiber (flax, hemp and kenaf) composites have been predominantly used in automotive interior panels, such as doors, pillar trim, trunk liners and package or rear-parcel trays.

The tensile strength of glass fibre reinforced composites is approximately 38 MPa and agave based composites can replace glass fibre composites especially for non-load bearing applications. In this study, parcel trays were manufactured from agave and polypropylene blend nonwovens. Agave and polypropylene fibres in the ratio 50/50 were processed to form needle punched nonwovens. The nonwovens exhibited an areal weight of ± 400 g/m2. Four layers of the nonwoven mat were placed in a 1160 x 660 mm mould and compression moulded at 200^{o}C for 2 minutes (Figure 9a). Polyester lining material was placed on top of nonwovens and compression moulded to form the finished product (Figure 9b). Currently studies are going on to compare properties of agave fiber based parcel trays with that of conventional glass fibre products.



(a)

(b)

Figure 9: Parcel tray from agave and polypropylene nonwovens

5. Conclusions

The study focused on the use of agave fibre blends as reinforcements in polypropylene composites. In terms of mechanical strength, the incorporation of agave nonwovens improved the tensile strength of the composites. Composites containing agave-polypropylene (A-PP) nonwovens exhibited superior mechanical properties compared to the other two. The storage modulus of the composites was found to be maximum for agave-PP composites indicating increased stiffness in composites containing synthetic fibres. Damping was found to decrease with incorporation of agave nonwovens. This was attributed to lowering of molecular mobility in reinforced composites. Parcel trays for automotives were developed from agave-PP nonwovens by the process of compression moulding. Future studies will focus on comparing the cost – performance properties of agave based parcel trays with that of glass fibre based products.

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CAPTIONS TO FIGURES

Figure 1: Pictures of (a) A-PALF, (b) A-PP and A-WW nonwovens

Figure 2: Variation of tensile strength with composites

Figure 3: Variation of modulus with composites

Figure 4: SEM micrographs of (a) A-PALF (b) A-PP (c) A-WW composites

Figure 5: Variation of flexural strength of composites

Figure 6: Variation of storage modulus with temperature of composites

Figure 7: Variation of tan delta with temperature of composites

Figure 8: Thermograms of composites

Figure 9 (a) and (b): Parcel tray from agave and polypropylene nonwovens

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