

Interface modification in jute/polypropylene composites

Naturfaserverstärkte Polymere haben in Abhängigkeit von ihren Grenzschichteigenschaften unterschiedliche mechanische Eigenschaften. Viele Untersuchungen fokussieren auf eine Faserflächenbehandlung. Unsere Untersuchungen berichten über den Effekt von maleinsäureanhydridgepfropftem Polypropylen (MAHgPP) als Haftvermittler in Jute/PP-Verbunden, wobei zwei Matrices (PP1: hohe Molmassen, PP2: niedrige Molmassen) verglichen werden. An kurzfaserverstärkten Spritzgieß-Prüfkörpern wird das mechanische Verhalten untersucht. Unter Einbeziehung der an Einzelfaser-Modellverbunden ermittelten Grenzflächeneigenschaften wird eine modifizierte Mischungsregel formuliert, die sehr gut die experimentellen Ergebnisse beschreibt. Ein Zusatz von 2 Ma-% MAHgPP kann die Adhäsionsfestigkeit und folglich die mechanischen Eigenschaften der Composites wesentlich verbessern. Wir fanden, dass die Zugfestigkeit der Jutefasern im Gegensatz zu anderen Verstärkungsfasern proportional zu deren Querschnittsfläche ansteigt.

1 Introduction

Natural fibre reinforced thermoplastics have a good potential as a substitute for wood-based material in many applications. The development of environment-friendly *green* materials is because of natural fibre's biodegradability, light weight, low cost, high specific strength compared to glass and carbon, recycling and renewing natural sources. The jute fibre is an important bast fibre and comprises bundled ultimate cells, each containing spirally oriented micro-fibrils bound together. The primary component of the fibre is cellulose, which is a linear condensation polymer consisting of D-anhydroglucopyranose units joined together by β -1,4-glucosidic bonds [1]. The major part of the cellulose consists of a microcrystalline structure with high order of crystalline regions. Other components of the jute fibre are hemicellulose, lignin, pectin, waxy and water soluble substances. Because of the structural features, the high level of moisture absorption and poor wettability of the natural fibre material results in insufficient adhesion between fibres and polymer matrices leading to debonding during use and aging [2,3]. Previously, selective removal of non-cellulosic compounds constitutes was the main objective of the chemical treatments of natural fibres to improve the performance of fibre reinforced composites. Various fibre surface treatments have been reported, such as: silane, alkali, combination of alkali and silane, monomer grafting under UV radiation, maleic anhydride grafted polypropylene, and others. A comprehensive literature overview is given in our latest paper [4].

Among those of using commercial coupling agent maleic anhydride grafted polypropylene (MAHgPP) has been found to be the most efficient in improving interfacial adhesion of natural fibres and a PP matrix. Overall, the MAHgPP's ability to enhance the composite properties depends on many factors, such as type of MAHgPP (graft level, random or block copolymer, molecular weight), miscibility of MAHgPP with the PP matrix, PP grade, composite processing conditions.

Theories for both elastic strength and modulus of composites have

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been well developed. The rule of mixtures (ROM) concept has been most widely used for strength and modulus predictions although it is not completely adequate for composites containing short, randomly oriented fibres [5]. A large number of research interest was dedicated to theoretical and numerical models with varying degrees of success. However, agreement with the experimental data of randomly oriented short fibre reinforced composites cannot be accurately predicted with these models, since these models disregard the existence of an interphase [6]. Therefore, an extended approach is developed in our recent work based on the established procedures for getting reliable information on properties of the interphase [7].

The objective of our work was to find out how matrix modification based on MAHgPP affects the interfacial adhesion and mechanical properties of jute/PP composites. Both micro-mechanical methods and mechanical composite testing are used to evaluate the introduced properties changes in two kinds of polypropylenes PP1 (low melt flow rate) and PP2 (high melt flow rate) matrixes. Interfacial fracture morphology is studied by atomic force microscopy (AFM). In addition, single fibre tensile test, moisture absorption, and fibre length/diameter distribution analysis were also used to assess the specific behaviour of jute fibres in PP-composites.

2 Experimental

Jute yarn of 1100 tex yarn fineness was obtained from Spinnerij Blancquaert NV (Lokeren, Belgium). PP1 was supplied by Borealis A/S as commercial grade HD 120 M and PP2 was provided by Basell (Frankfurt, Germany) as commercial grade Purell 570 U. The commercial grade Exxelor PO 1020 (Exxon Mobil Corp., USA) was selected on base of comparison with different commercial MAHgPP grades performed previously [4,6]. The material properties are listed in Table 1 in terms of densities, average molecular weight, melt flow rate (MFR), melting points, prices and maleic anhydride graft level of MAHgPP.

Properties	Jute	Coupling agent	PP1	PP2
Density at 23 °C [g·cm ⁻³]	1.4	0.9	0.908	0.910
Melt flow rate [g/10 min]	–	125 (190°C/1.2kg)	8 (230°C/2.16kg)	92 (230°C/2.16kg)
M _w [kg·mol ⁻¹]	–	86	290	145
Melting point [°C]	–	160	180	170
Maleic anhydride graft content [wt-%]	–	0.5-1	0	0
Price [€·kg ⁻¹]	0.3	4.5-5.0	-	0.77-0.79

Table 1:
Material parameters of fibre, matrices and coupling agent

Composite samples of jute yarn, PP granules, and MAHgPP granules were compounded by a co-rotating twin-screw extruder ZSK 30 (Werner & Pfleiderer, Stuttgart, Germany) with optimised screw configuration [4]. The extruded strands were cooled by immersion in a water bath and pelletized.

From compound granules, the dog-bone shaped specimens (160 x 10 x 4 mm³, according to DIN 53455, specimen No. 3, ISO 527.2) and plates (80 x 80 x 2 mm³) were made by injection moulding (Ergotech 100, Demag Ergotech Wiehe GmbH).

The tensile strength of the filaments was determined in accordance with EN ISO 53812 and ASTM D 1577. The measurement was conducted using the Fafegraph ME testing device (Fa. Textechno) after having determined the fineness of each selected fibre using a vibration approach in a Vibromat testing equipment (Textechno).

PP was removed from jute/PP composites (granules, plate) by boiling in xylene (135 °C) for 14 h. Fibres were spread between a glass slide and a cover plate. The lengths and diameters of extracted fibres were measured by the analySIS Software program (Soft Imaging System GmbH, Münster, Germany) using semi-automatic two points measuring mode. It should be noted that the cross-section of jute fibres is oval or polygonal, however it was adapted to a circular one in this work. About 600 data points were collected for each fibre dimension distribution.

Mechanical testing was done using the universal testing machine Zwick 1456, the tensile and un-notched Charpy impact tests were performed according to ISO 527-2 and ISO179/1eU, respectively.

For single-fibre pull-out tests, firstly the model micro-composites were prepared by using a institute-made embedding apparatus with embedding lengths of 50-500 µm. The pull-out test was carried out with identical pull-out velocities (0.01 µm·s⁻¹) at ambient temperature. From each force-displacement-curve the maximum force F_{max} , and the embedded length l_e were determined. The apparent adhesion strength τ was calculated, according to $\tau = F_{max}/(d \cdot \pi \cdot l_e)$.

The fibre diameter d was measured microscopically at two perpendicular views and five different positions along the fibre for each viewing angle. The pulled-out fibres also were collected for AFM investigation. Each fibre/PP combination was evaluated by about 15 to 20 single tests.

Fracture surfaces of the fibres after pull-out tests were examined using an AFM (a Digital Instruments D3100, USA) in tapping mode at resonant frequency of approximate 300 kHz with a drive amplitude of 200 mV.

3 Results and discussion

3.1 Fibre mechanical properties

We first characterised the natural fibre tensile properties. The statistical variability of the tensile strength of brittle materials and natural fibres is commonly described by following Weibull statistic analysis:

$$P = 1 - \exp \left[- \frac{V}{V_0} \cdot \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (1)$$

$$V = S \cdot L_f \quad (2)$$

where P is the cumulative probability of failure [$i/(n + 1)$] at the tensile stress σ . The parameters V , V_0 , S , L_f , are tested volume, scaling constants, mean cross-sectional area and fibre length, respectively. The parameters m and σ_0 are the Weibull shape factor (or modulus, slope of the distribution) and the scale factor (or characteristic strength) of fractured fibres. To determine Weibull modulus, 200 jute fibres were tested at gauge lengths of 5, 10, 15, and 20 mm. As expected, the tensile strength of natural fibres in the fibre direction depends on the fibre length and exhibits considerable

scatter (Table 2), due to the statistical distribution of flaws such as pits and nodes [8] and not uniform cross-section of fibre. Nevertheless, the specific tensile strength (strength/density) of jute fibre is similar to that of glass fibres [580 MPa/(g·cm⁻³)] and highlights the application for lightweight composites.

Gauge length [mm]	Tensile strength ± s.d. [MPa]	Specific tensile strength [MPa/(g·cm ⁻³)]	Weibull distribution parameters	
			Weibull-modulus m	Charact. strength σ ₀ [MPa]
5	770 ± 243	550	2.9	743
10	629 ± 228	449	2.6	611
15	610 ± 198	436	3.3	601
20	582 ± 223	416	2.8	577

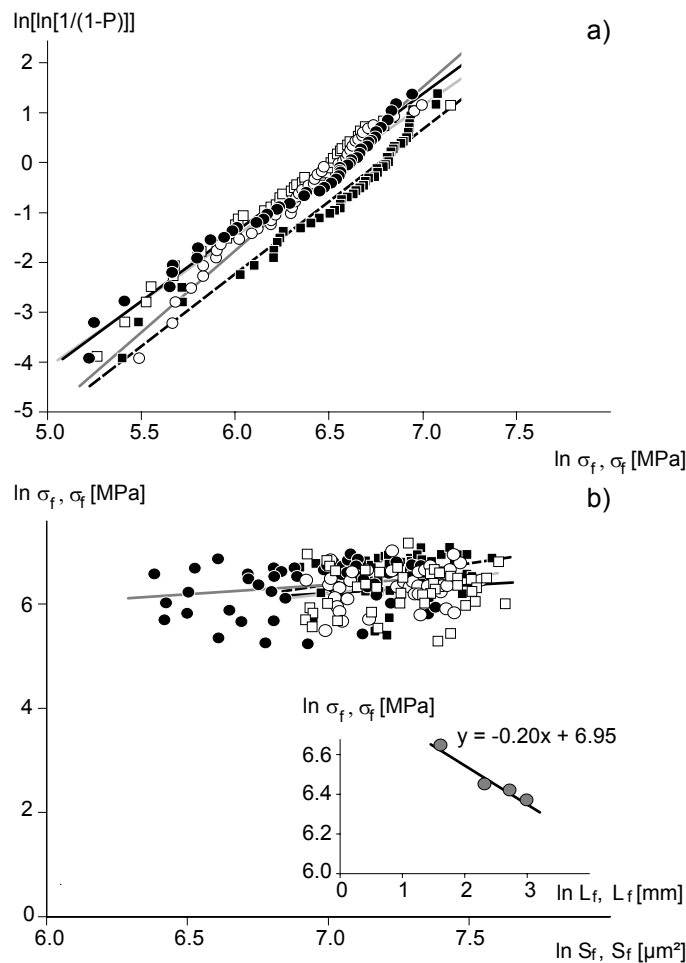
Table 2: Tensile strength of jute fibres (s.d. = standard deviation)

Fig. 1a: Weibull plots of fracture probability as a function of jute fibre tensile strength for different gauge lengths: gauge length:

5 mm (---) $y = 2.90 \cdot x - 19.67$;
 10 mm (●) $y = 2.61 \cdot x - 17.16$;
 15 mm (○) $y = 3.25 \cdot x - 21.26$;
 20 mm (□) $y = 2.77 \cdot x - 18.02$

Fig. 1b: Variations of jute fibre tensile strength and mean tensile strength as a function of fibre cross-section area and gauge length (the insert), respectively. The data were fitted using the least squares method.

5 mm (---) $y = 0.53 \cdot x + 2.96$;
 10 mm (●) $y = 0.39 \cdot x + 3.68$;
 15 mm (○) $y = 0.44 \cdot x + 3.34$;
 20 mm (□) $y = 0.21 \cdot x + 4.85$



As shown in the Weibull plots of Fig. 1 and Table 2, the fibre tensile strength tend to decrease with increasing fibre length from 5 mm to 20 mm. This result is as expected, since the probability of critical defects causing fracture increases with increasing length. The Weibull modulus calculated using the strength distribution (Fig.1a) for four fibre lengths is in the range of 2.6 to 3.3 mm. On the other hand, the Weibull modulus can also be determined from the fibre length dependence of fibre mean strength (Fig. 1b insert), however, it is about 5 mm. This is substantially higher than the Weibull modulus measured from the distribution of tensile strength. It is not commonly studied the effect of fibre cross-section area variability on the measured Weibull modulus. Generally speaking, a

fibre with larger cross-section area should have greater chance to have a bigger flaw, therefore, it is weaker than that with smaller cross-section area. For natural fibres, there is a distribution of individual fibre cross-section area, shape within both a roving and individual single fibre, so that the tested volume will vary even at a fixed gauge length. Thus, the measured strength distribution will be created by the overlap of the distribution of strength due to the volumetric distribution of flaws and due to the variable fibre volume. Because of this effect, the natural distribution of fibre strength will be artificially broadened, reducing the measured Weibull modulus from the distribution of tensile strength.

Notably, the measured tensile strength actually increased with jute fibre cross-sectional area at a constant fibre length despite the scatter (Fig. 1b). This is inconsistent with the general statistic failure behaviour for brittle materials, where the strength tends to decrease with increasing volume because of an increase of the probability of critical defects. This finding suggests that the intrinsic tensile properties of jute fibre is proportional to fibre cross-sectional area associated with its growth time. It is reported that jute fibre has a cellulose content of more than 60 % and a micro-fibril angle in the range of 7°-12° to the fibre axis [8]. These structure characters are strongly maturity dependent and in turn, affect significantly the overall mechanical properties of natural fibres. However, the Weibull theory assumes a random distribution of flaws within the volume of the samples with same properties. Thus, it is clear that the tensile strength of the natural fibre has been grossly oversimplified in the previous commonly used Weibull model.

3.2 Interphase and composite mechanical properties

An effective use of fibre strength is dependent on both the interfacial adhesion properties and the critical fibre length. The micro-mechanical evaluation was done by using single fibre pull-out test. It is well known that fibre length plays an important role in the mechanical performance of fibre reinforced composites. Using the Kelly-Tyson theory, the critical fibre length, defined as the minimum fibre length where the maximum allowable fibre tensile stress can be achieved, is roughly calculated as:

$$l_c = \frac{\sigma_f \cdot d}{2 \cdot \tau} \quad (3)$$

where σ_f is the fibre strength at gauge length equal to the critical fibre length and τ is the apparent interfacial shear strength.

Therefore, investigations of the effect of coupling agents on the interfacial properties of jute fibre/PP composites have been considered with two kinds of matrices (PP1 and PP2).

Table 3 shows an increase of apparent interfacial shear strength 91 % for PP1 and 68 % for PP2 while using 2 wt-% MAHgPP. Interestingly, the τ values of 19.8 MPa and 16.3 MPa for the Jute/PP1 and Jute/PP2 with 2 % MAHgPP are very close to the shear yield strength values of the pure PP1 and PP2 matrix as calculated from the VonMises criterium, being 19.8 MPa and 19.0 MPa (tensile strength values drawn from Fig. 2), respectively. This results indicated that the good interaction between jute fibres and the PP matrix with MAHgPP was achieved and the composites could be used effectively in the strength applications. Importantly, we also calculated l_c values, using the experimental data of τ and σ_f value from the fit line in the insert of Fig. 1b, which are in the region of 670-1117 μm for different systems.

Table 3:
Experimentally measured apparent adhesion strength, calculated fibre tensile strength at the corresponding critical fibre length, and fibre orientation factor (η), s.d. = standard deviation

Samples	$\tau \pm \text{s.d.}$ [MPa]	σ_f [MPa]	l_c [μm]	η
Jute / PP1	10.3 ± 1.5	1028.3	1074.6	0.18
Jute / PP1+2-wt% MAHgPP	19.8 ± 2.7	1130.1	670.2	0.36
Jute / PP2	9.7 ± 1.0	1020.3	1117.2	0.10
Jute / PP2+2-wt% MAHgPP	16.3 ± 3.1	1096.8	778.1	0.28

In order to examine in detail the effect of coupling agents on the interfacial properties, we use the natural fibres which have the mean fibre length (ca. 200 μm) significantly shorter than the above calculated critical fibre length. Therefore, the fracture behaviour of bulk composites can be dominated by fibre/matrix interfacial debond and matrix failure without extensive fibre breakage. It should be reminded that significant damage to the glass and other man-made fibres may occur in a conventional injection moulding process, resulting fibre length lost. To confirm if variation of the jute fibre dimensions exists during composite process, measurements of the fibre length, L_f , and diameter, d , were performed before (compound granules) and after (composite plates) injection moulding process. Since the great scatter usually determined of natural fibres, the values of fibre dimensions for each system are a rough approximation based on more than six hundred individual tests. Table 4 summarizes the average fibre lengths and diameters derived from compound granules and plates. Of note is that no significant changes in the fibre geometrical dimensions were observed after injection moulding, suggesting that no long fibres and bundles were cut into shorter ones or separated into elementary cells during the processing. Therefore, jute fibres are rather wear resistant during injection moulding.

Table 4:
Average fibre length (L_f), diameter (d), and length/diameter ratio (L_f/d) derived from compound granules and plates (s.d. = standard deviation)

Samples	$L_f \pm \text{s.d.}$ [μm]	$d \pm \text{s.d.}$ [μm]	L_f/d
Granules	243 ± 162	23 ± 15	10.6
Plates	244 ± 154	22 ± 12	11.1

Fig. 2 shows the tensile strength and modulus of jute/PP composites affected by the coupling agent. It is interesting to see that increasing tensile strength with increasing fibre content is only valid for the systems with the coupling agent (Fig. 2a). Using 2 wt-% MAHgPP as matrix modification increased the tensile strength of jute/PP composites of about 41 % for PP1 and 28 % for PP2 at the same fibre volume content. For the systems without coupling agent, the fibre acts as an included filler in the resin matrix, which actually weakens the composite because of poor interfacial adhesion. As aforementioned, the measured average fibre length in the short-fibre composite plate is far below the critical fibre length for all the systems in this study.

This allows us to use a modified *rule of mixture* taking account of interfacial properties for the strength of natural fibre/polymer composites, σ_c , as a function of volume fraction [7].

$$\sigma_c = \left(\frac{\eta \cdot L_f \cdot \tau}{d} - \sigma_m \right) V_f + \sigma_m \quad (4)$$

where η is the fibre orientation factor which has values between 0 and 1. σ_m and V_f are tensile strength of matrix and volume fraction of fibre, respectively. Fitting the equation to the tensile values of various systems in Fig 2a and using the fibre parameters and interfacial adhesion data in Table 3 and 4, gives values of the fibre

orientation factor in the region of 0.10 to 0.36 and an average value of 0.23 (Table 3). This value is very close to the theoretical value for a random arrangement, $\eta = 0.20$ [10] or 0.23 [11]. The significant variation of tensile strength for different systems indicates fibre alignment is not the only factor which affects mechanical performance; interfacial adhesion and how the fibre influences matrix properties also have a significant effect. Clearly, these theoretical lines based on above equation correlated well to the experimental results, demonstrating the accuracy of the 'rule of mixture' theory, taking into account of interfacial adhesion properties, applied to natural fibres in PP matrix.

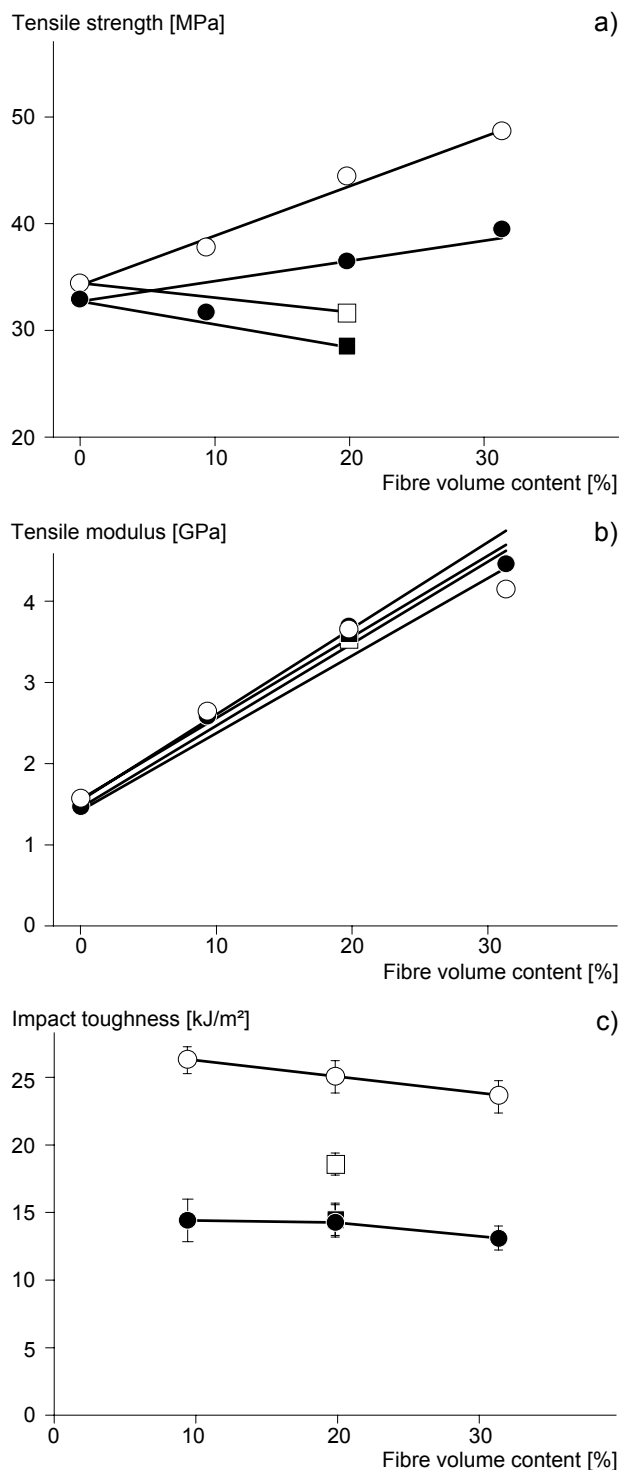


Fig. 2: Influences of interfacial adhesion on the mechanical properties of jute/PP composites. The theoretical linear regression lines and the corresponding fibre orientation factor, η , and the efficient stress transfer parameter, β , are based on the Eqs. 4 and 5, respectively:

a) Tensile strength as a function of the fibre volume content: jute/PP1 + 2 wt-% MAHgPP, $\eta = 0.36$ (○); jute/PP2 + 2 wt-% MAHgPP, $\eta = 0.28$ (●); jute/PP1, $\eta = 0.18$ (□); jute/PP2, $\eta = 0.10$ (■)

b) Modulus as a function of the fibre volume content: jute/PP1 + 2 wt-% MAHgPP, $\beta = 0.42$ (○); jute/PP2 + 2 wt-% MAHgPP, $\beta = 0.49$ (●); jute/PP1, $\beta = 0.51$ (□); jute/PP2, $\beta = 0.51$ (■)

c) Charpy impact toughness as a function of the fibre volume content. Error bars represent the standard deviation: jute/PP1 + 2 wt-% MAHgPP composite (○); jute/PP2 + 2 wt-% MAHgPP composite (●); jute/PP1 composite (□); jute/PP2 composite (■)

Fig.2b gives a comparison of the variation in the experimental and theoretical tensile modulus values of the jute/PP composites with fibre volume fraction. According to the Hirsch model which is a combination of parallel and series *rule of mixtures* models [12], the Young's modulus of the composite is determined by the equation:

$$E_c = \beta \cdot (E_m \cdot V_m + E_f \cdot V_f) + (1 - \beta) \frac{E_f \cdot E_m}{E_m \cdot V_f + E_f \cdot V_m} \quad (5)$$

where β is a factor of efficient stress transfer between fibre and matrix. E is the Young's modulus; c , m , f , refer to the composite, matrix and fibre. From the figure, it is clear that the model can adequately fit to the data. For all systems, E_c values increase almost linearly with increasing fibre content. In contrast to above tensile strength, the tensile moduli are not affected by the coupling agents. Generally, Young's modulus reflects the capability of both fibre and matrix material to transfer the elastic deformation in the case of small strains without interface fracture. Therefore, it is not surprising that the tensile modulus is less sensitive to the variation of interfacial adhesion than the tensile strength which is strongly associated with interfacial failure behaviour.

Unlike the significant improvement of the tensile properties with increasing fibre content, the impact toughness decreases slightly (Fig. 2c). It is attributed to a change from ductile to brittle fracture behaviour with increasing fibre content. Besides, the probability for fibre agglomeration [13] also increases at higher fibre content, creating regions of stress concentration that require less energy to initiate or propagate a crack [14]. The important toughening mechanism in fibre reinforced polymer composites is crack-bridging by fibres associated with frictional sliding during fibre pull-out, which in general be affected by the interfacial adhesion. Due to the improved stress transfer effect by the coupling agent, the impact toughness for jute/PP1 matrix composite increased by about 40 %. However, the impact toughness of PP2 matrix composites was not significantly affected by the coupling agent. It is believed that the deformation behaviour of PP is strongly dependent on its molecular weight. As demonstrated by above tensile test (Figs. 2a,b), the PP2 having lower molecular weight shows both lower strength and modulus values than the PP1 having higher molecular weight. It seems that the smaller molecular weight of PP2 might promote polymer fracture rather than the deviation of the major crack path away from the polymer to interphase region, resulting less extensive to interfacial debonding. Consequently, it reduces the sensitivity of interfacial adhesion contributions to toughness.

Further insight can be obtained from the failure surface observation. The representative AFM microphotographs (Fig. 3) are apparently different for jute fibres before and after pull-out tests. Fig. 3a shows that the rough surface topography of as-received jute fibre (elementary fibre with diameter ca. $22\mu\text{m}$) consists of many small and distinct stripes being dominant on its surface, which is made up of cellulose microfibrils bound together by a matrix of lignin and hemicellulose [15]. Such fine surface texture features on the fibre surface can not be observed after the fibre being pulled out from the PP matrix (Fig.3b), suggesting the fibre surface was well coated with the matrix material. In other words, cohesive failure occurs and the polymer very near the fibre appears to have been extensively stretched during fibre pull-out.

It is evident that the matrix modification by 2 wt-% MAHgPP improved the interaction between fibres and matrices. The MAHgPP

coupling agent is able to act as a compatibiliser for polar natural fibre and non-polar polymer matrix systems. A hypothetical model of the interface between MAHgPP with hydroxyl groups of jute fibre is shown in Fig. 4. The strong interfacial adhesion between the fibre and MAHgPP treated PP matrices can be understood from this model, in which likely both chemical (ester bond) and physical interactions (hydrogen bond) should be formed between the cellulose fibre and coupling agent. The PP chain of MAHgPP diffuses into the PP matrix involved interchain entanglements. On the other hand, the maleic anhydride group forms both covalent and hydrogen bonds with the hydroxyl groups of the fibre. These cause better adhesion between the fibre and the matrix. Therefore, the transfer of stress from the matrix to the fibres is improved and lead to higher tensile strengths.

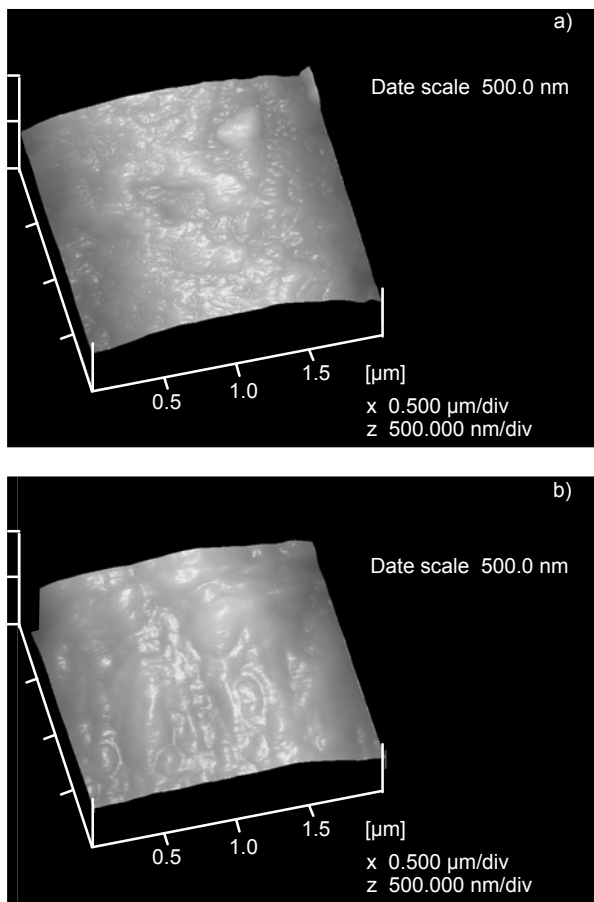


Fig. 3: AFM topographies of jute fibre surface as received (a) and pulled out fracture fibre surface from PP1 with 2 wt-% MAHgPP (b)

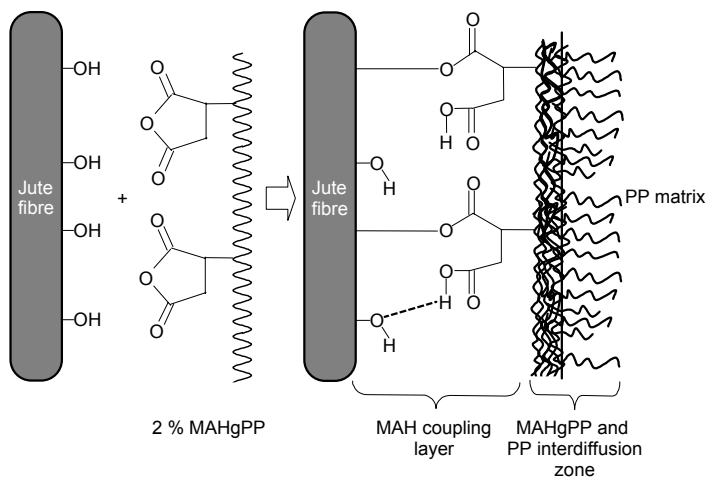


Fig. 4: Hypothetical structure of MAHgPP coupling agent and jute fibre at the interface

4 Conclusion

The addition of 2 wt-% MAHgPP to PP matrices significantly improved the adhesion strength with jute fibres and in turn the mechanical properties of composites. The PP-grade significantly affected the improvement of Jute/PP composite by MAHgPP coupling agents. Higher molecular weight PP with less melt flow rates improved the mechanical properties to a greater extent than lower molecular weight. The tensile modulus of jute/PP composites showed less sensitive the variation of interfacial adhesion. Taking into account the interfacial properties, a modified *rule of mixture* (ROM) theory was formulated which fits well to the experimental tensile strength results.

The measured fibre tensile strength actually increased with jute fibre cross-sectional area at a constant gauge length, which is inconsistent with the statistic failure behaviour of other man-made fibres or brittle materials where the probability of critical defects causing fracture increases with increasing volume. The intrinsic tensile properties of jute fibres are proportional to fibre cross-sectional area associated with its growth time. The previous commonly used Weibull model does not describe the tensile strength of the natural fibre when the cross-sectional area is considered.

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