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EFFECT OF DIFFERENT ANHYDRIDES ON MECHANICAL PROPERTIES OF AGRO-WASTES AND NOVOLAC RESIN COMPOSITES

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Wheat straw (WS), sorghum straw (SS), Cane bagasse fiber (CBF) and teak saw dust (TS) composites were prepared separately with novolac resin in 50:50 (w/w) ratio. These fillers were treated with and without maleic anhydride (MA), phthalic anhydride (PA) and succinic anhydride (SA). Anhydrides treatment improves tensile strength, young's modulus, flexural strength, and flexural modulus of the composites over the untreated filler composites. The order of increment in tensile strength and Young's modulus is recorded as SA > PA > MA for CBF, while for WS and SS order is MA > SA > PA. In case of TS the order of increment in tensile strength and Young's modulus is SA > MA > PA. Flexural strengths of CB and SS are recorded as SA > MA > PA. MA > SA > PA is the order of flexural strength and flexural modulus of TS and WS. Amongst three anhydrides, PA treatment shows least effect while MA and SA treatments are more effective. The difference in their effectiveness is due to their reactivity as well as the desorption of anhydrides by the fillers.

INTRODUCTION

Mechanical properties are dependent of degree of bonding between filler and matrix. In order to evaluate the percentage of surface area of filler that has covered by resin inexpensive optical evaluation method is reported [1]. Albritton [2] reports effect of resin coverage and distribution on resin efficiency in medium density fiberboard. The relationship between resin coverage, resin distribution, and resin efficiency of medium density fiberboard as affected by blending variables are discussed [2,3]. With increase in amount of resin the fiber coverage increases and in turns the mechanical properties increase. It is found that with thermoset resins the mechanical properties of lignocellulose filled composites increases. As the polar lignocellulose filler tends to react with thermoset resin while secondary

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crosslinking diminishes the interfacial gap and results into unique infusible composite structure [4].

Hydrophobic nature of binding matrix and hydrophilicity of filler material in case of wood polymer composites result in to phase separation and hence appear detrimental to mechanical properties. Either, use of coupling agent or treatment to filler material is the way to get rid of this problem. Mechanical properties improved with treatment of fiber by urea formaldehyde, dimethylene urea and dimethylolhydroxy-ethylene urea in the presence of inorganic salt has been reported [5]. Modification through reaction with melamine formaldehyde and phenol formaldehyde resin is also reported [6].

Effects of chemical treatment on mechanical properties of agro-fibers like sisal, hemp and banana is reported [7]. Comparative study of different anhydride treatments with reference to mechanical properties proved the compatibilisation ability of anhydrides [8]. All the reports describe increased compatibility due to surface modification of lignocellulose fiber.

In present work the effect of MA, SA and PA is studied on mechanical properties of wheat straw, sorghum straw, cane bagasse fiber and teakwood sawdust with novolac resin composites.

EXPERIMENTAL

Phenol formaldehyde resin was synthesized using acidic pH. Details of resin synthesis are given elsewhere [4]. All the fibrous filler materials were treated using MA, PA and SA for the period of 2 h. Treated and untreated fibers were mixed with novolac resin using 50:50 weight ratio. Details of filler treatment and composite preparation are given in our earlier research paper [8]. Mechanical properties including Young's modulus, tensile strength, flexural modulus and flexural strength were calculated using UT-2303 (R&D Electronics, Mumbai). The samples of dimension $100 \times 10 \times 5$ mm were used. The test was carried out at 25°C with a crosshead speed of 25 mm/min.

RESULTS AND DISCUSSION

Effect of Anhydride Treatment on Tensile Strength and Young's Modulus of CBF Filled Novolac Composites

Figure 1 shows the trend of tensile strength and the Young's modulus for CBF filled novolac composites. The reactivity trend observed was as $\text{SA} > \text{MA} > \text{PA}$. As treatment of anhydride to lignocellulose filler implies ester linkage as well as acid groups by reacting with surface $-\text{OH}$ groups,

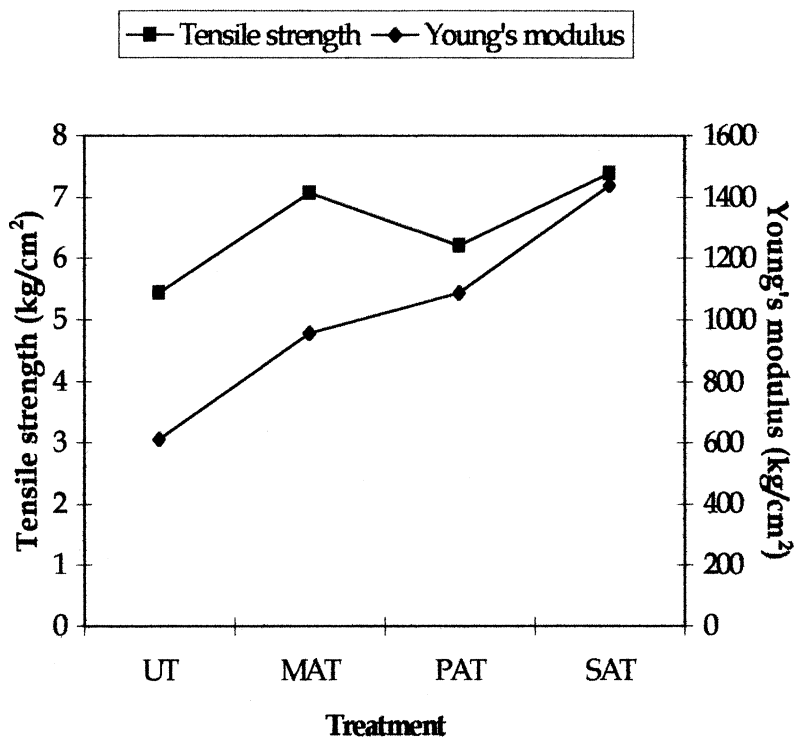


FIGURE 1 Effect of anhydride treatment on tensile properties of CBF filled Novolac composites.

it affects the stress transfer. More is the reactivity higher is the frequency of chemisorption and desorption. Sugarcane bagasse has been reported to contain 48% of cellulose [9]. Hence maleation proceeding with moderate reactivity tends to impart moderate acid as well as ester linkage. This enhances the compatibility moderately. The extent of esterification achieved through maleation has lower value. It failed to impart higher modulus and decreased the modulus value as compared to untreated filled composites.

Phthalialation proceeding with gradual speed having lowest frequency of chemisorption and desorption amongst three of the anhydrides and provided sufficient ester linkages, necessary to impart efficient stress transfer and efficient load compensation. Succination having higher reactivity imparted ester linkage in extra to the acid group produced which hinders lower modulus value to the composite. Hence imparted proper stress transfer.

Effect of Anhydride Treatment on Tensile Strength and Young's Modulus of SS Filled Novolac Composites

From Figure 2, it is observed that the filler responded towards all the anhydride treatments employed for its surface modification and improved the tensile properties of composites. Maleiation imparted the highest tensile strength to the composite followed by the composite containing succinated SS as filler. But phthaliation treatment results least improvement amongst three anhydrides. However the trend in improvement in Young's modulus is MA > PA > SA. As un-dissolved SA used to dissolve continuously till the completion of treatment reaction, here in the chemisorption and desorption phenomenon get controlled as compared to maleiation. Higher wax content and lower ratio of ligno-cellulose to total carbohydrate content restrict the surface modification. Insufficient amount of ligin to criss-cross the cellulose available completely also affect the synergy between filler and matrix. While in experimentation it was observed that the treatment reaction of any anhydride to SS using xylene as reaction medium imparted reddish brown color to xylene along with

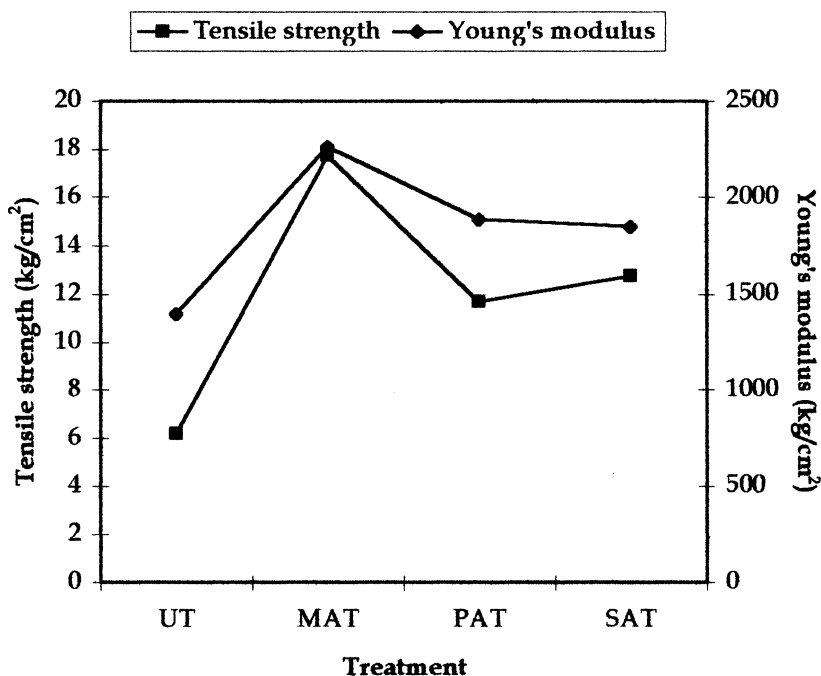


FIGURE 2 Effect of anhydride treatment on tensile properties of JS filled novolac composites.

liberated waxy chemicals. Hence, this unexpected and uncertain trend of response to various anhydride treatments by SS may be attributed to the density and higher wax content.

Effect of Anhydride Treatment on Tensile Strength and Young's Modulus of TS Filled Novolac Composites

Figure 3 shows that the tensile strength of composite increased with treatment by anhydride. The anhydride treatment has remarkable effect in improvement of tensile strength of untreated TS filled composites after treatment.

The improvement in tensile strength after maleiation was of approximately 33%. The same was of 80% in case of succinated TS filled com-

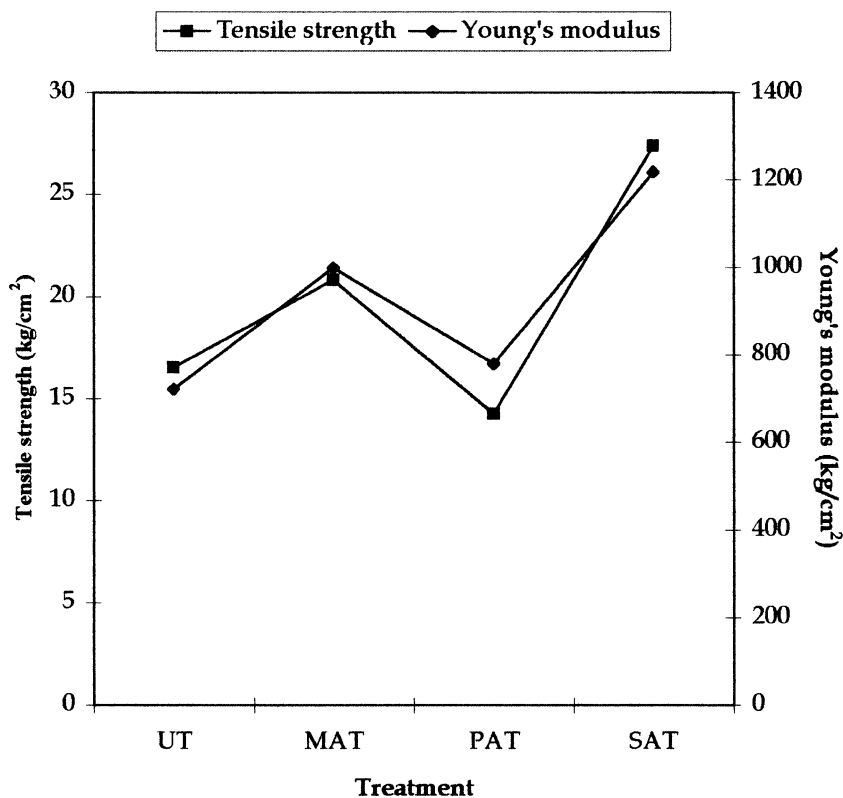


FIGURE 3 Effect of anhydride treatment on tensile properties of TS filled novolac composites.

posite. However the phthaliation is less effective due to its bigger molecule which is not excessive with the lignocellulose material of TS.

Effect on Tensile Strength and Young’s Modulus of Different Anhydride Treated WS Filled Novolac Composites

It is very clear from Figure 4 that the anhydride treatment improved tensile strength of 600-μ mesh WS filled novolac composites. The maleiation imparted the highest tensile strength whereas succination showed less tensile strength than maeation, while phthaliation exhibited least strength amongst three anhydrides. Like tensile strength the young’s modulus also showed an improvement by anhydride treatment of WS. But SA treatment showed highest improvement on flexural modulus while PA and MA showed more or less similar improvement. Surface treatment increases probability of cross-linking between lignocellulose filler and novolac resin. Itself the

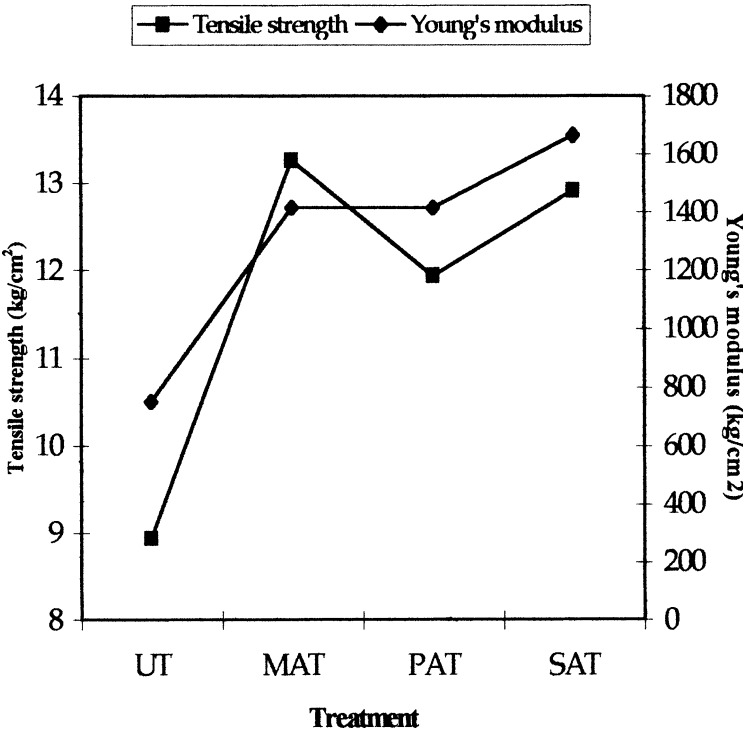


FIGURE 4 Effect of anhydride treatment on tensile properties of WS filled composites.

hydroxyl group from lignocellulose filler tends to react with novolac by bond formation.

Effect on Flexural Strength and Flexural Modulus of Different Anhydride Treated CBF Filled Novolac Composites

Figure 5, illustrates that three of the treatment reagents have capacity to improve flexural strength by increasing the compatibility between CBF and novolac. Amongst three of the anhydrides PA showed least improvement on flexural strength due to ring structure present in its structure. The ring structure also imparted stiffness and hardness to the composite prepared.

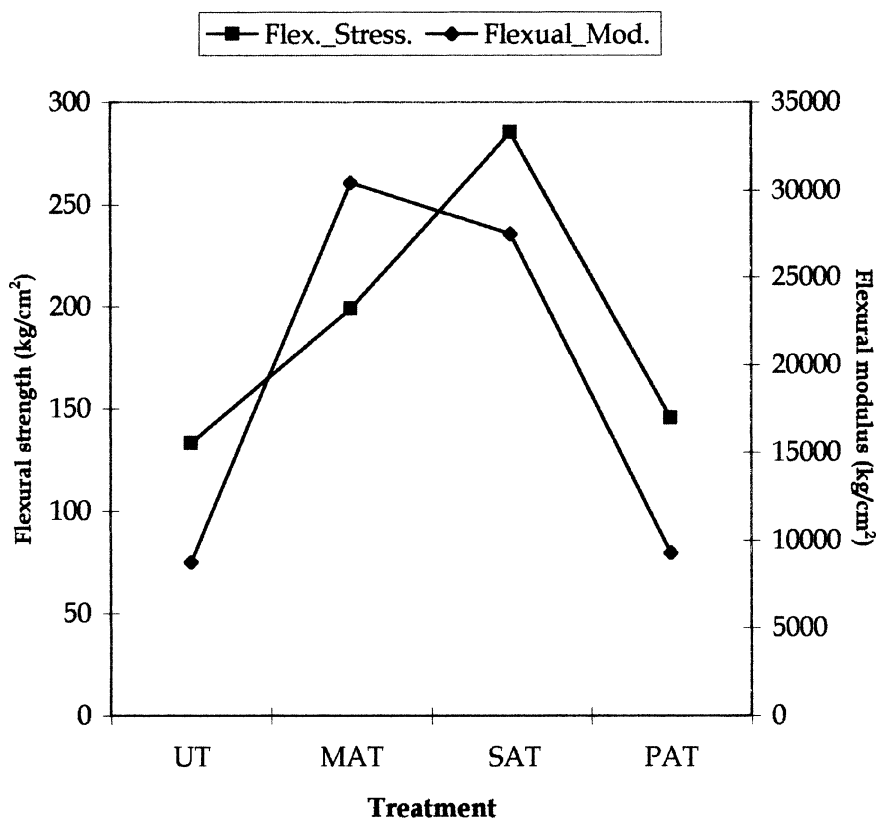


FIGURE 5 Effect of anhydride treatment on flexural properties of CBF filled novolac composites.

Maleiation imparted higher flexural strength. Reactivity of MA was lower than other anhydrides. However, the double bond present in its structure facilitated the shearing capacity of the modified fiber units with each other and helped orientation of all the inter-linked chains of natural polymer.

The remarkable effect of maleiation on flexural modulus was exhibited by attributing the value of 30430 kg/cm². SA though reacts up to higher extent yet the frequent chemisorption and desorption of ester linkages diminished the modulus. Desorption results in increased concentration of acid groups on to surface of lignocellulose filler. These acids tend to interfere with the cross-linking between matrix and filler. In case of phthaliation the increased proportion of ring structured bulky group decreased flexural modulus by imparting hardness to the composites.

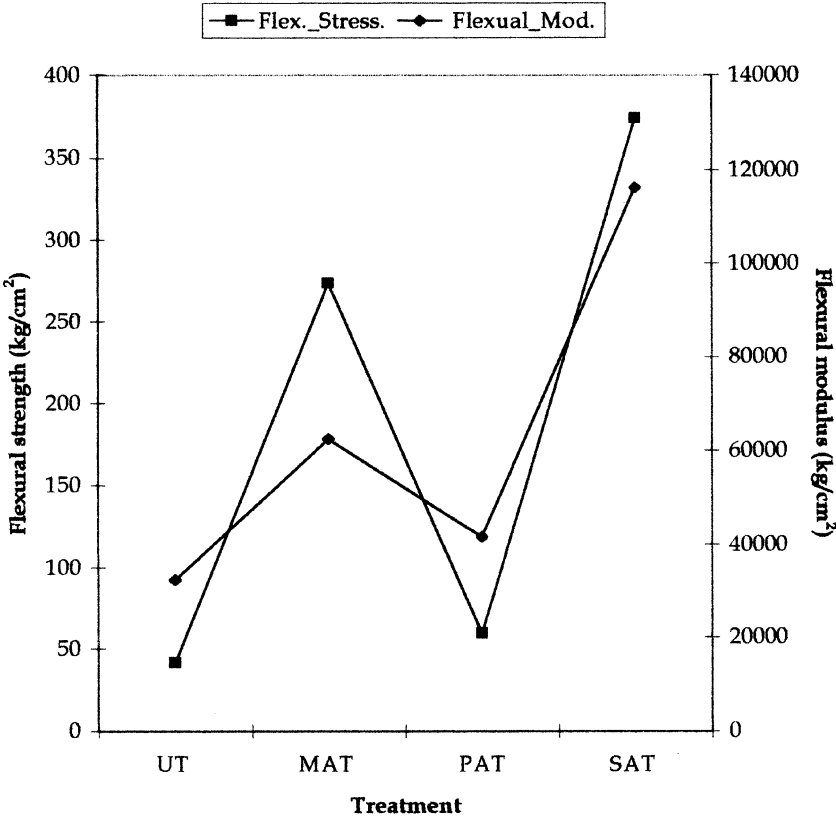


FIGURE 6 Effect of anhydride treatment on flexural properties of JS filled novolac composites.

Effect on Flexural Properties of Anhydride Treatment on SS Filled Novolac Composites

Figure 6 shows that three of the anhydrides affected the flexural strength with their peculiar manner of reactivity. SA reacted up to highest extent as un-dissolved anhydride for given concentration (1% in xylene) employed to modify filler surface continued its dissolution with respect to consumption of dissolved anhydride regularly. Thus imparts the highest flexural strength and maximum modulus to the composite. Ring structure present in the PA imparted hardness to the filler which on combination with novolac, -ring structured matrix resin, made the composite tough. Hence phthaliation imparts less flexural properties. MA with the help of double bond structure imparted good flexural strength to the fiber. But lacking behind in provision of extra ester linkage as compared to SA attributed flexural strength and modulus 275 kg/cm^2 .

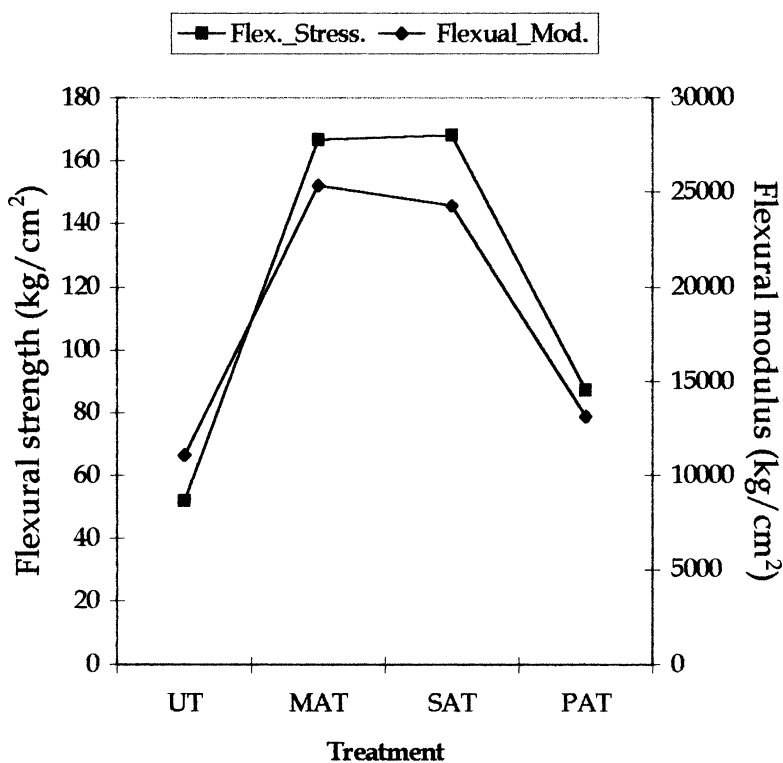


FIGURE 7 Effect of anhydride treatment on flexural properties of TS filled novolac composites.

Effect on Flexural Strength and Flexural Modulus of Different Anhydride Treated TS Filled Novolac Composites

Effect of maleiation proved by exhibition of the highest flexural strength by maleited 850- μ TS incorporated composite (Fig. 7). PA has tendency to impart hardness after treatment/esterification hence imparted the lower flexural strength. Succination imparted moderate flexural strength. Succinated TS filled composite performed with better flexural strength achieved through higher degree of extra esterification. More is the cross linking present in between filler and matrix of composite prepared higher is the shear strength of backbone chains present as interpenetrating network of cross-linked bonds.

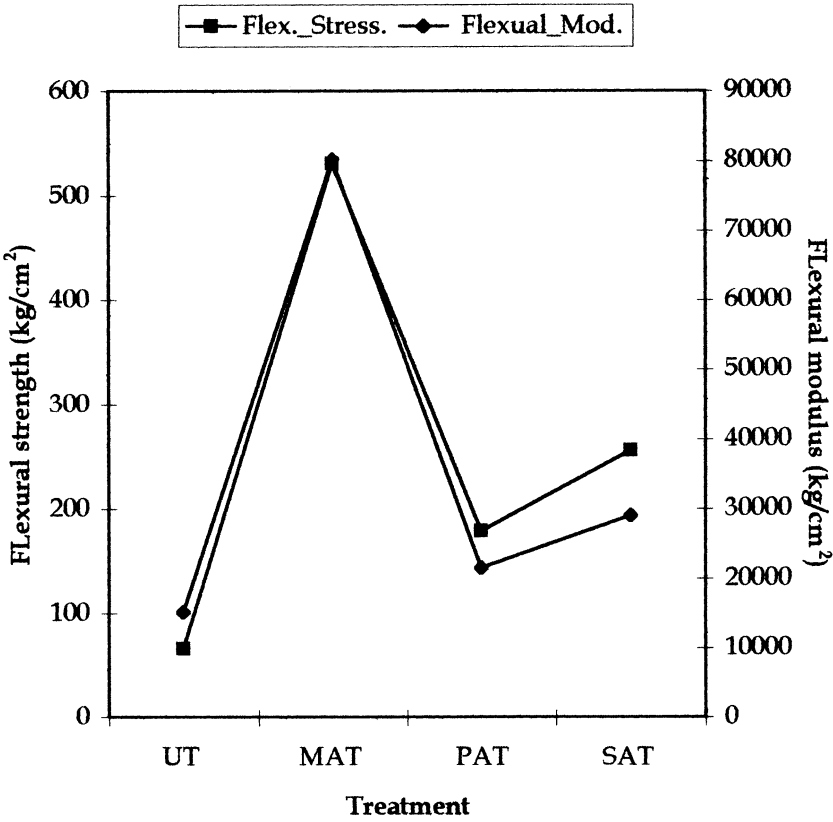


FIGURE 8 Effect of anhydride treatment on flexural properties of WS filled composites.

In correlation with the flexural strength of respective anhydride treated TS incorporated novolac composites, the flexural modulus was exhibited by the composites. TS, being dense and compact particulate filler imparted hardness to its composites and made it tough. However, with well-established grain orientation in its structure TS facilitated efficient stress transfer easily.

Effect on Flexural Strength and Flexural Modulus of Different Anhydride Treated WS Filled Novolac Composites

Three of the anhydrides tend to provide higher flexural strength to composites by increasing compatibility between matrix and filler through esterification of lignocellulose filler surface (Fig. 8). Phthaliation imparted hardness to the filler by introducing ring structure that was reflected by lowered flexural strength and modulus. Maleiation provides higher flexural strength and modulus than that of phthalated filler incorporated composites. Succination imparted the maximum flexural strength and modulus to composites. Trend of efficacy of different anhydrides to provide flexural properties to WS filled novolac composites in ascending order was MA > SA > PA.

CONCLUSION

The SA, MA and PA treatment improve the tensile strength, Young's modulus, flexural strength and flexural modulus of CBF, SS, TS and WS based novolac composites. However the variation in properties is observed amongst different anhydride treatments on different fillers. It is due to the internal structure i.e chemical constitution of lignocellulose (ratio) of fillers and reactivity of anhydrides with fillers.

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