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Surface Characterization of Heat-Treated Wood Filled Styrene Maleic Anhydride (SMA) Composites

Isıl İşlemli Odun Dolgu Stiren Maleik Anhidrit (SMA) Kompozitlerinin Yüzey Karakterizasyonu

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Abstract

The main objective of the present study was to evaluate the effect of thermal treatment on the surface properties of heat treated wood flour filled Styrene Maleic Anhydride (SMA) composites. SMA is an engineering polymer in the automotive for interior and exterior construction applications by injection molding and thermoforming. The heat-treatment of the pine wood was performed at 212 °C for 8 h. The SMA polymer was filled with untreated or thermally treated wood flour from 0 to 50 wt%. After the extrusion process by twin screw extruder, the specimens were produced by injection molding machine. As a results, the surface roughness values of the filled SMA composites increased with increasing with heat treated loading filler. Similarly, the wettability of heat treated and untreated wood flour SMA composites increased with increasing content of the wood flour.

Keywords: Wood thermoplastic composite, Heat treatment, Wettability, Surface roughness

Öz

Çalışmanın ana hedefi, ısıl işlemli odun dolgulu Stiren Maleik Anhidrit (SMA) kompozitlerinin yüzey özellikleri üzerine ısıl muamelenin etkisinin değerlendirmesidir. SMA, enjeksiyon kalıplama ve ısı ile şekillendirme ile iç mekan ve iç mekan uygulamaları için otomobil sektöründeki mühendislik polimeridir. Çam odununa ısıl işlem 212 °C'de 8 saat süre ile uygulanmıştır.. SMA polimerlerine ağırlıkça %0'dan %50'ye kadar ısıl işlemli ve ısıl işlemsiz odun unu katılmıştır. Çift vidalı ekstrüzyon kalıplama işleminden sonra, örnekler enjeksiyon kalıplama makinesinde üretilmiştir. Sonuçlara göre, ısıl işlemli dolgu oranı arttıkça SMA kompozitlerinin yüzey pürüzlülüğü değeri artmaktadır. Benzer olarak, odun dolgu oranı arttıkça, ısıl işlemli odun dolgulu SMA kompozitlerinin ıslanabilirliği artmaktadır.

Anahtar Kelimeler: Odun termoplastik kompozit, Isıl işlem, Islanabilirlik, Yüzey pürüzlülüğü

1. Introduction

WPCs as a material are obtained from wood or similar lignocellulosic fibres, plastics and additives. Plastic or polymer material used in WPC is usually recycled or waste plastic: either thermoplastic (like polypropylene or polyethylene) or thermoset (like phenol plastic or urea formaldehyde). The ratio of wood, plastic and additives in WPC depends on the manufacturing process and desired properties of the end-product, but usually the amount of wood in WPC is in the range of 20-85 % (Koto and Tiisala 2004, Mali and Rautiainen 2005). The formulation, including the contents of wood, plastic and additives, can significantly affect the properties of wood-plastic composites (Wolcott 2003, Lu et al. 2000, Caulfield et al. 1998, Stark and Rowlands 2003). Several research efforts about using recycled waste wood fibres materials and plastics to produce value-add, recyclable, and environmental friendly products were also published (Hwang et al. 1994, English et al. 1994).

Styrene maleic anhydride (SMA) copolymer is a thermoplastic polymer obtained by the copolymerization of styrene and maleic anhydride monomers and can be preferred as heat stabilizer, coupling agent and compatibilizer. The SMA is an engineering polymer in the automotive for interior and

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exterior construction applications by injection molding and thermoforming. It can also improve the reaction and adhesion between polymer and fillers in the composite industry (Bledzki et al. 2002, Sperber 2002, Riedel and Nickel 2003). When compared to conventional wood based composites, such as medium density fiberboard (MDF) and particleboard (PB), is quite limited to interior and exterior applications (moist application) due to the having strong ability to absorb water. Despite the hygroscopic nature of natural fibers, the hydrophobic thermoplastic matrix decelerates the uptake of water, and durability of WPCs has significantly been improved when compared with used decking (Clemons and Ibach 2004, Morrell et al. 2006, Lomelí-Ramírez et al. 2009).

Thermal modification improves the durability of wood (Hill 2006, Rowell 2007, Ayrilmis et al. 2011). Previous studies reported that thermal modification reduce wood's hygroscopicity, increase the degree of crystallinity of cellulose, and improve dimensional stability and resistance to fungal degradation (Bhuiyan et al. 2000, Kandem et al. 2002, Rowell et al. 2009, Stanzl-Tschegg et al. 2009, Windeisen et al. 2007, Pfriem et al. 2010). Several wood researchers have used heat treatment to improve wood properties (Hillis 1984), increased mass loss and color changes (Hakkou et al. 2006), and decreased some mechanical properties, as well as the wettability of wood (Esteves and Pereira 2009). Wettability is important for good adhesion in wood bonding and to establish contact between molecules of wood and adhesive (Wood Handbook 2010). The wettability of wood can be characterized by contact angle analysis. This analysis is very significant to define the adhesive and coating properties of wood and wood based composite surfaces (Petrissams et al. 2003). There is no information on surface properties of the SMA containing different amounts of thermally treated wood flour. The main objective of the present study was to evaluate the effect of thermal treatment on the surface properties of heat treated wood flour filled SMA composites.

2. Materials and Method

2.1. Materials

The SMA (XIRAN® SE700) was supplied by Poyscope Polymer, USA. It has a density of 1.08 g/cm^3 (Maleic anhydride contents 10% wt., melt flow 22 g/10min at 240°C/10.0 kg). The eastern white pine (*Pinus strobus* L.) was used as fillers in this study were kindly supplied by Wicks Lumber in Pittsfield, ME, USA.

2.2. Heat Treatment

Pine wood samples were cut from the sapwood of a radial board of eastern white pine (*Pinus strobus* L.). Cubic samples with 360 mm x 20 mm x 20 mm were cut with clear faces, kept in a conditioned room at 20°C and 50% relative humidity for 3 weeks and weighed afterwards. The heat treatment was made in an oven heated by electric coils located in the walls and with exhaustion of the heated gases by natural convection through an opening in the oven wall. The heat-treatment of the pine wood was performed at 212 °C for 8 h. The treatment started by putting the samples at ambient temperature in the oven. The time to reach the treatment temperature was about 60 min. After heat treatment, the solid wood board samples were removed from the oven and ground in a grinder. Wood flour of pine greater than 60 mesh was used as raw materials to prepare the wood flour/SMA composites. Untreated samples were used as the control.

2.3. Sample Preparation

The untreated wood flour, heat treated wood flour and SMA were dried to a moisture content of less than one percent using an oven at 105°C for 16-h. The matrix polymer, SMA, was mixed with the untreated wood flour, or heat treated wood flour. The compounding was conducted with a Brabender Prep-mixer equipped with a bowl mixer and the process temperature was measured in real time. The temperature was set at 230°C and rotor speed at 60 rpm. The untreated wood flour, heat treated wood flour were added to the mixer when the polymer appeared well melted. Mixing was done for 15 min until the torque stabilized. The SMA–wood flour compounds were granulated using a lab-scale grinder. The ground particles were dried in an oven at 105°C for 16 h before being injection molded into ASTM test specimens. All materials were injection molded using a barrel temperature of 230°C mold temperature of 230°C injection pressure of 2500 psi. The compositions of the composites are shown in Table 1.

2.4. Determination of Contact Angle (Wettability)

An imaging system was used to measure contact angle of water droplets for the prepared specimens. Contact angle measurement was done in 15-s intervals from 5 to 25 s for wetting behavior. For convenient observation, image profiles on each test specimen exposed at 15 s were measured to reveal its isotropic behavior. For contact angle, six water droplets on each specimen were observed along and across the grain direction, respectively. The images were captured

Table 1. Composition of SMA, Untreated and Heat Treated Composites

SMA (wt%)	Untreated wood-UT (wt%)	Heat treated wood-T (wt%)
100	-	-
90	10	-
80	20	-
70	30	-
60	40	-
50	50	-
90	-	10
80	-	20
70	-	30
60	-	40
50	-	50

using the video camera. All captured images were then stored as image files and measured using SigmaScan® software (Figure 1).

2.5. Determination of Surface Roughness

Three roughness parameters characterized by ISO 4287 standard (1997), respectively, average roughness (R_a), mean peak-to-valley height (R_z) and maximum peak-to-valley height (R_y) were considered to evaluate the surface properties of the wood filled SMA composites. The surface roughness of the samples was measured with a sensitivity of 0.5 ηm. Measuring speed, pin diameter and pin top angle of the tool were 10 mm/min, 4 ηm and 90˚, respectively. The surface properties roughness parameters were calculated from the digital information.

3. Results and Discussion

3.1. Wettability

As shown in Table 2, the wettability of the wood flour SMA composites significantly increased with increasing loading filler. The samples containing the 50T (81. 11˚ for 15 s) had the highest wettability, while the lowest wettability was found for samples containing the 20UT (50. 69˚ for 15 s) among the all groups. The contact angle of the composites was significantly affected by increasing the wood flour loading content. This is expected because wood flour is a hydrophilic porous due to the presence of cellulose and hemicellulose polymers that are rich in functional groups such as hydroxyls, which readily interact with water molecules by hydrogen bonding associated with the SMA matrix has a hydrophilic nature and polar. Decreasing hydroxyl groups on the fiber surface as a function of the heat-treatment, hydrogen-bonding sites for water molecules decreased on the SMA composite surface. This resulted in a higher contact angle value for the specimens.

The surface activity of wood decreases the wettability of the wood. During the thermal modification process, the surface inactivation occurs. Surface inactivation is defined as a heat-induced change in the wood structure resulting in a loss of bonding ability (Trougton and Chow 1971). Northcott et al. (1962) confirmed that inactivation induced by heat treatment resulted in a decrease of the absorptivity of the wood. The effect occurs much more quickly at higher temperatures. An inactivated wood surface does not bond well with adhesives, because the inactivation process reduces the ability of an adhesive to properly wet, flow, penetrate and cure (USDA Wood Handbook 1999). A similar result was found in the present study. As the amount of the heat treat wood content in the wood/SMA composition, the wettability of the composites decreased, in other words contact angle values increased.

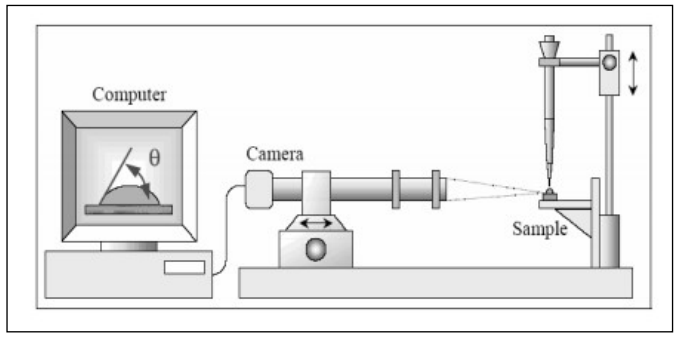

Figure 1. Scheme of contact angle determination device.

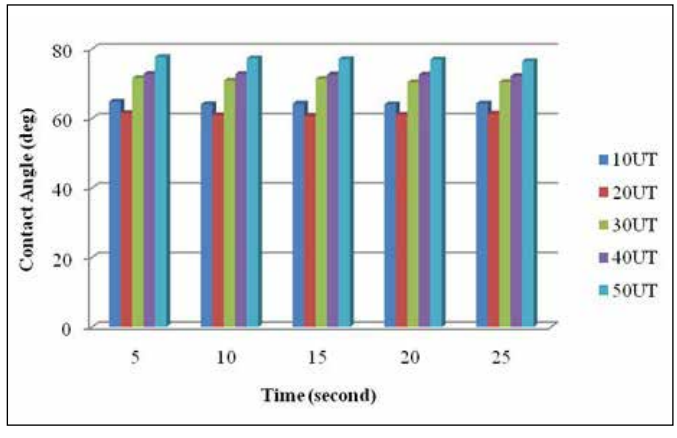

Figure 2. Effect of the wood flour loading on the wettability values of the wood flour SMA composites (**UT:** untreated wood SMA composite).

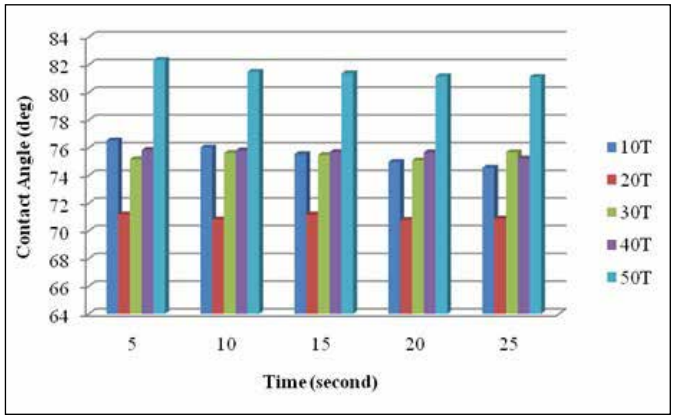

Figure 3. Effect of the loading flour on the wettability values of the heat treated wood flour SMA composites (**T:** treated wood SMA composite).

Table 2. Contact Angle Values Between Groups and Diffence (%)

Group Name	Contact Angle (deg at 15 s)	Difference (%)
10UT	64.36 (0.78)	17.35
10T	75.53 (0.32)	
20UT	60.69 (0.34)	17.26
20T	71.17 (0.19)	
30UT	71.38 (0.26)	5.73
30T	75.47 (0.24)	
40UT	72.69 (0.28)	4.12
40T	75.69 (0.52)	
50UT	77.13 (0.24)	5.50
50T	81.11 (0.49)	

**Values in parentheses are standard deviations.*

The wettability values of the wood flour SMA composites increased with increasing with heat treated wood flour. The samples containing the 20T had the highest wettability, while the lowest wettability was found for the samples containing the 50T.

3.2. Surface Roughness

The surface roughness of wood flour SMA composite samples was significantly affected by heat treatment. Significant differences among the wood flour SMA composites were determined ($p<0.05$) according to the ANOVA statistical analysis (Table 3). Homogeneity groups were determined individually for *Ra, Ry, and Rz* by Duncan's multiply range test.

The surface roughness of the samples significantly increased with increasing with loading filler and heat treatment. As the amount of the heat-treated wood content in the composites, the roughness values increased. The 10UT samples had the lowest average roughness with a Ra value of 0.94 (μm), while the highest roughness with a Ra value of 2.74 (μm) was found in the samples containing 50T. For example, when the heat treatment content from 30 wt-% to 50 wt-%, longitudinal direction average *Ra, Ry, and Rz* values of the samples increased by 16.10%.15.94%, and 13.73%, transversal direction average *Ra, Ry, and Rz* values of the samples increased by 32.95%, 37.90%, and 34.59%, respectively (Table 3).

The increases in the surface roughness of the wood flour/ SMA composites containing the heat treatment were mainly attributed to increasing volume percentages of the loading

Table 3. Effect of the Wood Flour Loading and Heat Treatment on the Surface Roughness (Ra, Ry, and Rz) Values of the Wood Flour SMA Composites

Group Name	Surface roughness parameters (μm)						Composite density ($g.cm^{-3}$)
	longitudinal direction			transversal direction			
	Ra	Ry	Rz	Ra	Ry	Rz	
10UT	0.94(A)	5.94(A)	4.20(A)	1.85(ABC)	10.69(ABCDE)	7.78(ABC)	1.06
10T	1.88(AB)	7.85(BC)	5.78(ABC)	1.28(ABC)	8.28(ABC)	5.74(ABC)	1.08
20UT	1.20(AB)	8.34(ABC)	5.30(AB)	1.44(ABC)	9.77(ABCD)	6.42(ABC)	1.10
20T	1.95(ABC)	11.95(ABCDE)	7.98(ABC)	2.50(BC)	15.67(CDE)	10.15(BC)	1.11
30UT	1.54(ABC)	9.79(ABCD)	6.80(ABC)	1.65(ABC)	11.13(ABCDE)	7.32(ABC)	1.13
30T	2.36(ABC)	14.68(BCDE)	9.61(ABC)	1.76(ABC)	11.74(ABCDE)	7.85(ABC)	1.12
40UT	1.58(ABC)	10.98(ABCDE)	6.95(ABC)	1.74(ABC)	12.07(ABCDE)	7.61(ABC)	1.15
40T	2.04(ABC)	12.6(ABCDE)	8.29(ABC)	1.85(ABC)	12.3(ABCDE)	8.01(ABC)	1.14
50UT	2.72(C)	17.97(E)	11.30(C)	2.34(ABC)	17.34(DE)	10.47(BC)	1.17
50T	2.74(C)	17.02(DE)	10.93(BC)	2.34(ABC)	16.19(DE)	10.70(BC)	1.16

filler used in the experiments. The increment in the surface roughness could be due to the fact thermal degradation of wood starts at 100˚C, while above 200˚C, structural damage, a change in the compounds making up the wood, and the production of degradation products in the gas phase occur. At above 140˚C, dehydration reactions commence, causing a decreasing the hydroxyl content; this increases the surface roughness with increasing temperature (Budakci et al. 2011).

4. Conclusions

These preliminary findings indicated that the surface characteristics properties of the heat treated woof filled SMA composites were significantly affected by the thermal treatment of the wood fibers. The results of the present study have shown that the surface roughness values of wood flour SMA composites increased with increasing with heat treated wood flour. Wettability strength between the untreated and treated wood filled SMA composites were negatively affected by the thermal treatment of the fibers. It can be included that decreasing in the hydroxyl groups on the fibers resulted in a lower wettability on the composite surface. Wettability and surface roughness of the composites can provide good information on their ability to bond. Further studies should monitor the contact angles for longer time periods to attain a better understanding of the effect of the treatment variables on the surface quality of the heat treated wood filled SMA composites.

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