EFFECT OF FIRE RETARDANTS ON SURFACE ROUGHNESS AND WETTABILITY OF WOOD PLASTIC COMPOSITE PANELS

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Surface roughness and wettability of flat-pressed wood plastic composites (WPCs) incorporated with various fire retardants (FRs) (5, 10, or 15% by weight (wt)) at 50 wt-% content of the wood flour (WF) were investigated. The most common FRs, zinc borate (ZB), magnesium hydroxide (MH), and ammonium polyphosphate (APP), were used in the experiments. The WPC panels were made from dry-blended wood flour (WF), fire retardant (FR) powder, and polypropylene (PP) powder with maleic anhydride-grafted PP (2 wt-%) formulations using a conventional flat-pressing process under laboratory conditions. The contact angle measurements were obtained by using a goniometer connected with a digital camera and computer system. Three roughness measurements, average roughness (R_a), mean peak-to-valley height (R_z), and maximum roughness (R_y), were taken from the WPC panel surface using a fine stylus tracing technique. It was found that the surface smoothness of the WPC panels decreased with increasing content of the FR powder while the wettability increased. The control WPC panel without the FR had the smoothest surface, followed by the WPC panels containing the MH, ZB, and APP, respectively.

Keywords: Contact angle; Fire retardant; Surface roughness; Wettability; Wood plastic composite

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INTRODUCTION

Wood plastic composites (WPC) represents one of the rapidly growing markets within the plastic and wood industries. WPCs represent an emerging class of materials that combines the favorable performance and cost attributes of both wood and plastics (Stark et al. 2010). Although commercially less important, the predominant technology to produce WPCs is extrusion to obtain endless profiles and injection moulding leading to 3-dimensional forms. Another possibility, which has only little been explored, is to produce WPCs on a flat-press. The advantage of the latter technology is that only a relatively low pressure level is required as compared to the extrusion and injection molding. The productivity of the pressing technology is much higher than that of injection molding and extrusion. The flat-pressed thermoplastic composites made by using a dry-blending method have a clear cost advantage (Jarusombuti and Ayrilmis 2011).

The use of conventional wood-based composites, such as particleboard and medium density fiberboard (MDF), is quite limited for exterior and moist applications, due to the strong tendency of such materials to absorb water. By contrast, flat-pressed WPC panels show a considerably reduced affinity towards water, compared to conventional wood-based panels, which is caused by their relatively high thermoplastic content. However, polyolefins, such as polypropylene (PP) and polyethylene, the most common plastics employed in WPCs, burn and drip in case of fire, leading to a very risky scenario. Thus, fire retardant (FR) agents must be employed to improve fire behavior of the WPCs (Stark et al. 2010; Ayrilmis et al. 2011a). The most common commercial fire retardants for the WPCs are ammonium polyphosphate, magnesium hydroxide, and zinc borate (Ayrilmis et al. 2011a).

WPC panels can be succesfully overlaid with decorative wood veneer sheets using a suitable adhesive. A recent study by Jarusombuti and Ayrilmis (2011) reported that WPC panels may be a competitor to the overlaid wood-based composites produced for interior and outdoor furniture applications. When the WPC panels are used as substrate for thin overlays, such as melamine impregnated papers, wood veneers, vinyl films; or liquid coatings, such as paint, varnish, and lacquer, their surface properties in terms of roughness and wettability play an important role in determining quality of final product. Previous studies reported that the FR treatments had a significant effect on surface properties of veneer-based wood composites, such as plywood and laminated veneer lumber (Ayrilmis et al. 2006, 2009a). Effects of the FRs on physical, mechanical, and fire properties of WPCs have been extensively investigated in previous studies (Sain et al. 2004; Stark et al. 2010; Ayrilmis et al. 2011a). However, there is no information currently available for the surface properties of the WPC panels containing the FRs. WPC panels containing the FRs are mostly used in indoor environments due to the fire safety requirements. As a result of this, the WPC panels containing the FRs as well as the ones without the FR are needed to finish with the surface coatings to enhance their esthetics. The study reported here, investigated effect of various FRs on the surface roughness and wettability of the WPC panels to be used as substrate for thin overlays or liquid coatings. The WPC panels were made using different formulations of dry-blended wood flour, PP, coupling agent, and FR powder using a conventional flat-pressing process under laboratory conditions.

EXPERIMENTAL

Materials

Commercial softwood WF (Jeluxyl WEHO 500V) used to produce to the WPC panel was obtained from a manufacturer (JELU-WERK) of WF located in Rosenberg, Germany. The WF was then dried in a laboratory oven at 102°C for 24-h to moisture content of 0-1% based on the oven-dry WF weight. PP powder (Moplen HP500V) (T_m = 163°C, ρ = 0.91 g/cm^3, MFI/230°C/2.16 kg = 120 g/10 min) produced by Basell Polyolefine GmbH (LyondellBasell Industries) in Wesseling, Germany, was used as the polymeric material. Maleic anhydride-grafted PP (MAPP) (Scona TPPP 8112 FA) (T_m = 155-170°C, ρ = 0.91 g/cm^3) powder was supplied by Kometra Ltd., Schkopau, Germany.

Three FR systems (powder) were investigated:

1) Ammonium polyphosphate (APP) (NH_4PO_3) (Exolit AP, 422, ρ = 1.9 g/cm^3, average particle size: 15 µm, Clariant Corp., Frankfurt, Germany)
2) Magnesium hydroxide (MH) ($Mg(OH)_2$) (Apymag 80S, ρ = 2.4 g/cm^3, average particle size: 3 µm Nabaltec AG, Schwandorf, Germany)
3) Zinc borate (ZB) ($3ZnO.2B_2O_3$), (ρ = 2.8 g/cm^3, average particle size: 10 µm) (Balmumcu Chemical Com., Istanbul, Turkey)

The chemicals used in the experiments were found to be safe even under the worst-case exposure assumptions by National Academy of Sciences (2002). The WPC panels incorporated with 5, 10, or 15 wt-% of the FR system had a WF content of 50 wt-%. It was also produced the WPC panels without the FR at 40, 50, 60, and 70 wt-% WF contents to compare to the WPC panels containing the FRs. Table 1 shows the formulation design of the WPC panels.

Table 1. Formulation Design of the WPC Panels

WPC panel formulation	WPC Panel Compositon							
	FR		WF		PP		MAPP	
	Volume (%)	weight (%)	volume (%)	weight (%)	volume (%)	weight (%)	Volume (%)	weight (%)
WF-40	0	0	24.6	40	60.0	58	1.8	2
WF-50	0	0	30.8	50	42.2	48	1.8	2
WF-60	0	0	36.9	60	33.4	38	1.8	2
WF-70	0	0	43.1	70	24.6	28	1.8	2
WF-ZB	1.4	5	30.8	50	37.8	43	1.8	2
WF-MH	1.7	5	30.8	50	37.8	43	1.8	2
WF-APP	2.1	5	30.8	50	37.8	43	1.8	2
WF-ZB	2.9	10	30.8	50	33.4	38	1.8	2
WF-MH	3.3	10	30.8	50	33.4	38	1.8	2
WF-APP	4.2	10	30.8	50	33.4	38	1.8	2
WF-ZB	4.3	15	30.8	50	29.0	33	1.8	2
WF-MH	5.0	15	30.8	50	29.0	33	1.8	2
WF-APP	6.3	15	30.8	50	29.0	33	1.8	2

FR: fire retardant, WF: wood-flour, PP: polypropylene,
MAPP: maleic anhydride-grafted PP, ZB: zinc borate, MH: magnesium hydroxide,
APP: ammonium polyphosphate

WPC Manufacture

The flat-pressed WPCs were manufactured using standardized procedures that simulated industrial dry-blend production at the laboratory. After mixing the WF, PP, MAPP, and FR powders, the mixture was placed in a rotary drum blender. Following the blending treatment for about 10 min, the mixture was weighed and then formed into a mat on an aluminum caul plate, using a 450 mm × 450 mm polymer based forming frame which is durable to high temperatures. Wax paper was used to avoid direct contact of the PP powder with the metal platens during heating and pressing. The mats were then subjected to hot-pressing, using a computer controlled press. The maximum press pressure, pressing temperature, and total press cycle were 45 N/cm^2, 210°C, and 500 s, respectively. At the end of the hot-pressing cycle, the panel was moved from the hot press into a press at room temperature for cooling. The resulting WPC panels were conditioned for one-week in the climate room having 65% relative humidity (RH) and 20°C before they were cut into test samples. Ten mm thick panels were then trimmed to a final size of 420 mm × 420 mm. A total of 39 experimental panels, three for each type of panel, were manufactured (Fig. 1). The average density value of the WPC panels was 800 kg/m^3.

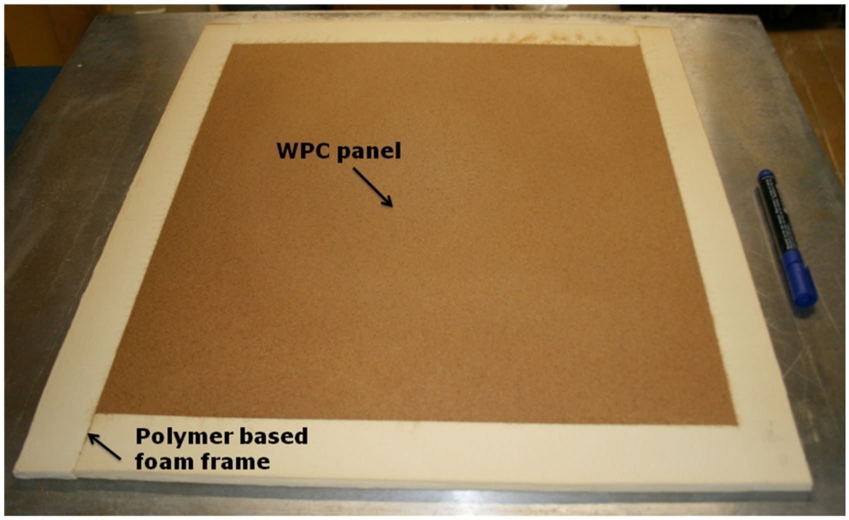

Fig. 1. A sample of the WPC panel containing the FR

Determination of the Surface Roughness

Currently there is no standard method to evaluate the surface roughness of WPCs. However, the stylus method is a well accepted one in the metal and plastic industries due to its accuracy and ability to provide well defined numerical values for the surface measured. Therefore, the surface properties of the samples were determined by employing a fine stylus profilometer (Mitutoyo SJ-301). The samples with dimensions of 50 mm × 50 mm × 10 mm were conditioned in a climate chamber at 20°C and 65% RH. Fifteen samples were used from each type of the panel for the surface roughness measurements. A total of sixty roughness measurements (four from each of fifteen samples: two measurements parallel and two measurements perpendicular to each other) were taken from each type of formulation.

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 WPCs. The surface roughness parameters were calculated from the digital information. The vertical displacement of the stylus was converted into electrical signals by a linear displacement detector before the signal was amplified and converted into digital information. R_a is the arithmetic mean of the absolute values of the profile deviations from the mean line and is by far the most commonly used parameter in surface finish measurement. The surface roughness of the samples was measured with a sensitivity of 0.5 µm. Measuring speed, pin diameter and pin top angle of the tool were 10 mm/min, 4 µm and 90°, respectively. The length of tracing line (L_t) and cut-off were 12.5 mm and 2.5 mm (λ), respectively. Measuring force of the scanning arm on the samples was 4 mN. Measurements were done at room temperature and pin was calibrated before the tests.

Determination of the Wettability

The wetting behavior of the WPC samples conditioned at 65% RH and 20°C was characterized by the contact angle method (goniometer technique). The contact angles were obtained using with a KSV Cam-101 Scientific Instrument (Helsinki, Finland). Using the sessile drop method, the most widely used procedure, the contact angle was determined simply by aligning a tangent with the sessile drop profile at the point of contact with the solid surface. The drop image was stored by a video camera and an image analysis system calculated the contact angle (θ) from the shape of the distilled water drop at room temperature. An imaging system was used to measure contact angle and shape and size of water droplets for the tested surfaces of the WPC samples. The image of the liquid drop was captured by a video camera, and the contact angle was measured by digital image analysis software of the KSV Cam-101 instrument. After the 5-µL droplet of the distilled water was placed on the sample surface, contact angles from the images were measured at 1 sec time intervals up to 60 sec total. If the contact angle (θ) is less than 90 degrees, the liquid is said to wet the solid. Fifteen samples with dimension of 50 mm x 50 mm x 10 mm were taken used from each type of formulation for the contact angle measurements. A total of sixty contact angle measurements, four from each of the fifteen samples were performed for each type of formulation.

Statistical Analysis

An analysis of variance, ANOVA, was conducted ($p< 0.01$) to evaluate the effect of the FR type and content on the surface roughness and wettability of the WPC samples containing the FRs. Significant differences between the average values of the WPC groups were determined using Duncan's multiple range test.

RESULTS AND DISCUSSION

Surface Roughness

The surface roughness of the WPC samples was significantly affected by the FR type and content. Significant differences among the WPC types were determined ($p<0.01$) according to the ANOVA statistical analysis (Table 2). Homogeneity groups were determined individually for R_a, R_y, and R_z by Duncan's multiply range test.

The surface roughness of the samples significantly increased with increasing FR content. The control samples (40 wt-% the PP) without the FR had the lowest average roughness with an R_a value of 2.7 µm, while the highest roughness with a R_a value of 6.4 µm was found for the samples containing 15 wt-% the APP, followed by the 15 wt-% the ZB (5.9 µm) and 15 wt-% the MH (5.6 µm) treatments, respectively. The R_a, R_y, and R_z values of the APP, ZB, and MH treatments at higher contents were always rougher than lower contents. For example, when the ZB content increased from 5% to 15 wt-%, the average R_a, R_y, and R_z values of the samples increased by 48%, 34%, and 52%, respectively.

The increases in the surface roughness of the WPC samples containing the FRs were mainly attributed to increasing volume percentages of the FRs used in the experiments. As shown in Table 2, the volume percentages of the FRs increased by 1.4 to 6.3%, depending on the FR type and content when the FR content increased from 5 to 15 wt-%. The volume percentages of the FRs in the WPCs differed from each other due to their different densities at the same content (wt-%) of the FRs. The surface roughness of the samples containing the APP was higher than samples containing the ZB or MH because the APP had the highest volume percentage among the treatment types. However, the surface roughness of the WPC samples containing the ZB was higher than that of the samples containing the MH in spite of the fact the volume percentage of the ZB was slightly lower than the MH at the same weight percentage of the FR. It was estimated that the smaller particle size of the MH (3 µm) caused to the lower surface roughness as compared to higher particle size of the ZB (10 µm).

Table 2. Surface Roughness and Contact Angle Values of the WPC Panels as a Function of Fire Retardant Type and Content

WPC panel type	Surface roughness parameters (µm)			Contact angle (θ) measuring intervals degree (°)			
	R_a	R_y	R_z	5 s	10 s	30 s	60 s
WF-40%	2.7 (0.2) A[1]	22.4 (2.8) A	13.2 (1.4) A	110.6 (9.8) A	109.4 (9.1) A	108.5 (9.4) A	108.1 (8.6) A
WF-50%	3.6 (0.3) B	28.3 (3.1) B	17.8 (1.8) BF	103.0 (8.3) B	102.2 (7.7) B	101.4 (7.1) B	100.7 (8.2) B
WF-60%	5.5 (0.4) CHI	38.6 (3.5) C	26.1 (2.2) CH	88.5 (7.7) C	87.1 (8.1) CH	86.2 (8.3) CI	84.5 (7.6) CHI
WF-70%	6.8 (0.4) D	46.8 (4.0) D	32.4 (2.8) DI	82.5 (7.6) D	80.3 (5.5) DI	79.2 (5.1) D	76.6 (4.8) D
WF-MH/5%	3.9 (0.2) B	30.3 (3.4) BE	17.3 (1.6) F	100.8 (8.0) B	99.7 (7.4) BE	98.4 (6.9) BF	97.1 (5.6) BE
WF-ZB/5%	4.0 (0.3) BE	31.7 (3.2) BEF	19.5 (1.7) EF	99.5 (7.5) B	98.2 (7.0) BE	97.0 (6.5) BE	95.5 (6.1) EF
WF-APP/5%	4.3 (0.2) EF	34.3 (2.9) EFG	20.6 (1.8) EFG	97.7 (6.8) BE	96.3 (7.3) EF	94.2 (7.0) EFG	93.4 (5.4) EFG
WF-MH/10%	4.7 (0.4) FG	35.6 (3.5) FG	22.8 (2.0) EC	93.5 (7.4) EF	92.6 (6.8) FG	91.8 (5.7) GH	91.0 (4.6) FG
WF-ZB/10%	4.9 (0.3) G	36.5 (3.6) FGH	24.0 (2.4) CG	92.2 (6.6) F	91.5 (7.1) CFG	90.0 (6.5) GC	89.4 (5.7) GC
WF-APP/10%	5.2 (0.4) H	38.8 (3.5) GHI	25.9 (2.7) CH	91.3 (6.9) F	89.4 (6.4) CGH	88.3 (5.6) CHI	87.6 (6.1) CH
WF-MH/15%	5.6 (0.5) HI	41.1 (4.0) HIJ	28.8 (3.2) HI	87.2 (6.2) D	86.2 (5.9) CHI	85.4 (6.3) CIJ	83.9 (4.9) HI
WF-ZB/15%	5.9 (0.6) I	42.5 (3.8) IJ	29.6 (3.0) HI	86.5 (5.7) CD	85.0 (6.0) HI	84.2 (6.1) DI	82.7 (5.4) HI
WF-APP/15%	6.4 (0.5) D	44.9 (4.2) J	31.3 (3.4) I	84.7 (5.5) CD	83.2 (5.2) I	82.5 (4.9) DJ	80.2 (4.5) DI

[1] Groups with same letters in column indicate that there was no statistical difference ($p<0.01$) between the samples according to Duncan's multiple range test. Values in the parentheses are standard deviations.

Particle size is one of the most important parameters affecting surface roughness of WPCs and wood-based composites. Previous studies reported that surface roughness of the WPCs and wood-based composites increased with increasing particle size (Ayrilmis et al. 2011b, Akbulut et al. 2000, Nemli et al. 2007). Ayrilmis et al. (2011b) reported that surface smoothness of the WPC panels (70 wt % WF and 30 wt % PP) significantly improved with decreasing the WF size. For example, WPC panels having a particle size smaller than 0.5 mm had the smoothest surface with an R_a value of 4.6 µm, while the roughest surface with an R_a value of 8.1 µm was found for the WPC panels having a particle size larger than 1 mm. A similar result was found in this study. The lower roughness of the samples containing the MH than the samples containing the ZB was attributed to the smaller particle size of the MH. This result was observed by inspection of the raw data from the surface roughness profilometer. The samples containing the MH showed recorded noticeably shallower ridges and valleys as compared to the samples containing ZB. The uniform distribution of the small FR partices on the surface layers decreased the surface roughness of the WPC. It also appears that as the FRs were added into the WPC panels their surface roughness increased due to not having well developed contact between the WF and PP on the surface layers.

The effect of the volume percentage of the WF on the surface roughness of the control WPC panels without FR is clearly shown in Table 2. When the volume percentage of the WF increased from 24.6% to 43.1%, the R_a, R_y, and R_z values of the WPC panels without the FR increased by 151%, 109%, and 145%, respectively. Pores and voids on the face layer of the WPC increased with increasing volume percentage of the WF. Coating systems need a substrate that permits primarily a mechanical link. The presence of the rough surfaces helps in the anchorage of the applied coating systems. A rough surface gives paints several possibilities to penetrate and create "fingers of resin", which helps in developing strong joins (Rolleri and Roffael 2010). Hence, the surface roughness is proposed to enhance intrinsic adhesion between WPC surface and coating by providing greater interfacial area and some mechanical interlocking mechanism. On the other hand, very high roughness has negative effects as high cost, due mainly to excessive volume of paint necessary to give surfaces smooth appearances.

The increase in the WF content resulted in significantly higher roughness on the face layers of the WPC samples without FR. This was mainly attributed to the anatomical structure of the wood particles, such as caves inside (vessels and cell lumens). The surface roughness of the WPC samples having a high content of the PP was lower than those of the samples having a low content of the PP. The lower surface roughness of the WPCs having a high content of the PP was due to the fact that plastics have the little interior void space and lower surface roughness as compared to the wood (Buehlmann et al. 2001). In addition, the lower surface roughness of the WPCs having a high content of the PP can be explained by the melting of the PP during the hot-pressing. When the PP powder in the WPC mat is melted by the press platens during the hot-pressing, it fills capillaries (micropores) in wood. The PP may crystallize on the wood particles and thereby wrapping wood particles better and leaving less exposed wood on the WPC surface. This results in lower roughness on the face layers of the WPC. However, the melting points of the WF and FRs used in the experiments are higher than the PP. For this reason, the WF and FR stay as powder on the face layers of the WPC during hot-pressing at 210°C. The increases in the surface roughness of the samples having higher contents of the WF and FR were also attributed to decreasing plastic content in the samples. As shown in Table 1, the plastic contents (by wt) of the WPCs decreased by 10-31% when the FR content increased from 5 to 15 wt-%.

Wettability

The wettability of the WPC samples significantly increased with increasing FR powder content. The samples containing the APP had the highest wettability, while the lowest wettability was found for the samples containing the ZB among the treatment types. The contact angle values of the samples significantly decreased with increasing volume percentage of the FRs. The APP had the highest volume percentage, while the ZB had the lowest volume percentage at all the treatment levels by weight. Inorganic FRs are known to make WPC more hygroscopic than WPCs without the FR. In particular, boron compounds and phosphates may have diverse effects on the hygroscopicty of wood (LeVan and Winandy 1990). The higher wettability of the samples containing the APP than the samples containing the MH and ZB was mainly attributed to the greater availability of the APP in the surface layers of the WPC panel. The APP's higher affinity for water was another reason for the increased wettability of the WPC surface (Ayrilmis et al. 2011a). In a previous study, it was found that WPC panels containing the APP had the highest thickness swelling and water absorption, followed by the WPC panels containing the MH and ZB, respectively (Ayrilmis et al. 2011a).

The decrease in the PP content of the WPC as a function of increasing FR content caused an increase in the wettability of the samples due to the hydrophobicity of the PP. Thus, the contact angle values of the samples containing the FR were lower than the control samples at the same content of the WF (WF-50). For example, the contact angles of the samples having 10 wt-% FR at 50 wt-% WF level were lower than those of the control samples having 50 wt-% WF (Table 2). However, when compared with the control samples having 60 wt-% WF, the samples having 10 wt-% FR and 50 wt-% WF had higher contact angles, namely lower wettability. This result showed that the wettability of the FRs used in the experiments was lower than the wood but higher than the PP. The wettability of the control samples increased with decreasing PP content. The face layers of the control samples with a high the PP content (58 wt-%) were less polar and thus repelled water, resulting in a lower wettability than in the case of the WPCs with a low PP content (28 wt-%). This was expected because wood is a hydrophilic porous composite of cellulose, lignin, and hemicellulose polymers that are rich in functional groups, such as hydroxyls, readily interact with water molecules by hydrogen bonding, whereas thermoplastic is hydrophobic and non reactive. Wood also has a critical surface energy in the 40–60 mJ/m^2 range (Gupta et al. 2007). On the other hand, the PP has very low surface energy (20-25 mJ/m^2), is hydrophobic, devoid of functional groups and develops smooth surfaces (Inagaki 1996). This large difference between the PP and wood causes the PP to be water repellent or hydrophobic.

A low contact angle is very important to capillary flow in the complex porous structure of wood to achieve a strong bond between adhesive and material surface. Jarusombuti and Ayrilmis (2011) reported that delamination strength between WPC surface and wood veneer sheet decreased with increasing contact angle value of the WPC. A similar result was also observed for wood based composite (Ayrilmis and Winandy (2009b). The strong correlations were found between the wettability and surface roughness of the WPCs at three levels of the FR treatment (Fig. 2). Liquid coatings such as paint, varnish, lacquer, and waterborne thermoset adhesives (aminoplasts) should adequately wet the WPC surface and establish physical adhesion. In addition, an increment in the wettability of the WPC is also important for the higher adhesion performance between the substrate and coating. The contact angle results showed that wettability of the WPCs was positively affected by increasing FR content. However, it should be considered that liquid coatings should be compatible with the FRs used in the WPC manufacure before they are applied to the WPC surface. Some liquid coatings can be sensitive to the chemical properties of the FRs.

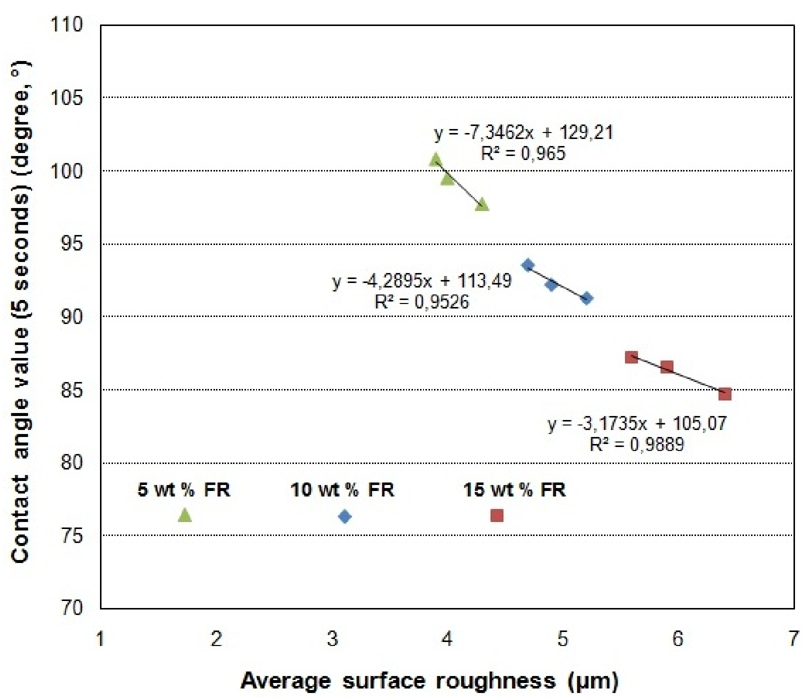

Fig. 2. Correlation between the surface roughness and contact angle of the WPC panels as a function of the FR content (wt %).

CONCLUSIONS

1. The surface smoothness of the WPC panels signficantly decreased with increasing FR content, while their wettability significantly increased. The volume percentage of the FR had more effect on the surface properties of the WPC than its weight percentage. The samples containing the APP had the highest roughness, followed by the samples containing the ZB and MH, respectively. The wettability of the control WPCs increased with increasing WF content while their surface smoothness decreased.

2. The surface roughness of the WPC panels increased with increasing particle size of the FR. Based on the roughness results, it may be said that when the FRs have the same volume percentages, the highest surface roughness for the WPC is obtained from the FR having the highest particle size.

3. The surface roughness and wettability of the WPC panels should be analyzed as a function of the volume percentage and particle size of the FR.

4. Some significant sectors for the overlaid/surface finished WPCs containing the FRs are building finishing and decoration, interior decoration, door, window, flooring, furniture, automotive interiors, and transportation (vehicles). With the industrial scope of the overlaid/surface finished WPCs expanding rapidly into applications in hospitals**,** kindergartens**,** schools, airport terminal lobbies, restaurant entertainment areas, office buildings, storage warehouses, the use of the fire-resistant WPCs is essential.

5. It is suggested that the WPC manufactures use modified paints or varnishes adapted to surface of the WPC containing the FR to prevent in-service adhesion problems.

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