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표면처리와 에이징이 고분자의 접착강도에 미치는 영향

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Effect of Surface Pre-treatments and Ageing on the Adhesive Strength of Polymer Joints

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Abstract: In this study, the effects of pre-treatment and ageing on the adhesive strength of polymer materials PE, PP and PVC are investigated experimentally. The experiments are carried out under mechanical abrasion, corona discharge and plasma methods at temperatures of -20, 0, 20 ºC and ageing for different times for 30, 60, 90 days at 50 ºC. The shear stress increased for PVC at -20 ºC, using both corona discharge and plasma methods. The shear strength of adhesively bonded joints reduced at an ageing temperature of 50 ºC and it continued to gradually reduce during ageing time from 30 to 90 days. The lowest strength was obtained at 50 ºC and 90 days' ageing.

Keywords: joint design, polymers, surface treatment, impact, ageing.

Introduction

Polymer materials have different problems in adhesion because of their low surface energy. The bond formed between surfaces during adhesion is directly related to its surface energy and the contact angle. In order to join the polymer materials and to obtain the desired strength, different surface treatments must be applied. Surface treatment can improve the surface energy and wettability. Wettability is an important notion to describe surface energy elevation. To describe the wettability, the surface of the drop and the surface of the material angle is examined. The degree of wettability which is requested can be provided by different surface treatments.[1,2]

The corona treatment systems working with electrical discharge at the polymer surfaces interact with the polymer molecules and free radicals. The radicals which are free and located on the surfaces of polymers can allow cross-link on surfaces with each polymer molecule. In corona, such as electrons and an electric field, ions are accelerated which forms a stream of charged particles. In this method, a polymer's high-frequency is exposed to a corona discharge generated by the high voltage alternating current.[3]

Plasma treatment is applied in a vacuum region. The polymer surface under reduced pressure is bombarded with an inert gas such as argon, helium or oxygen. On the plasma, surface molecules activated with noble gas ions increase the strength of the adhesive by crosslinking. In general, the concentration of functional groups added to a polymer surface by plasma may vary over time, depending on the ambient temperature and the temperature. Surfaces are reshaped in response to different circles. Surface shaping removes diffusion and polar function groups from the surface into the bulk of low molecular weight oxidized materials.[3,4]

Ku *et al.* [5] carried out research in order to improve the interfacial adhesion property between polypropylene/kenaf (PP/KF) fiber felt and polyurethane (PU) binder, and corona discharge treatment and grafted maleic anhydride were applied as a compatibilizer to PP/KF felt. In the case of modified PP/KF/

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PU composite treated by corona discharge and grafted maleic anhydride, the tensile and impact properties were improved.

Anagreh *et al*.[6] carried out research on low-pressure plasma pre-treatment of PPS surfaces for adhesive bonding. Surface treatment was carried out using an oxygen plasma technology on PPS. The results showed that oxygen plasma pre-treatment was gave the best adhesion for PPS surface rather than argon plasma.

Molitor *et al*.[7] used various methods of surface treatment including traditional treatments such as acid etch, anodization, novel plasma spray and laser treatments for both polymer composites and titanium. Sodium hydroxide etching gave the better strength and resistance. The best condition for two layer and bonding had been plasma treatment before bonding.

Dobbi *et al*.[8] studied the effects of plasma treatment on greenhouse PE cover aged under sub-Saharan conditions. The mechanical, physical and structural affects were analysed after applying plasma treatment. The results showed that changes from, the plasma treatment occurred on the surface.

Iqbal *et al*.[9] investigated the effects of atmospheric pressure plasma treatment on the surface energy of PEEK, CF and GF reinforced PPS. The polar surface has showed increasing surface energy of the sum of components. The atmospheric plasma treatment and low pressure plasma treatment were applied on the PEEK. The low pressure plasma treatment increased the surface energy more than the plasma treatment.

Ersoy and Kuntman[10] studied the surface properties of polymeric dielectric with a contact angle. Borax concentration was used for increasing the surface tension. Once the samples were immersed in an electrolyte solution for resistance of the dispersion liquid on the surface, this was shown to weaken the strength and tension properties. It was also observed that the contact angle of the polyester sample with borax contributions increased the surface energy.

Zhou and Havley[11] investigated whether microwaves were an efficient alternative energy source for polymer and composite productions using microwave technique. Microwaves can give good results for increasing surface energy. Microwave scans reduce the bonding time to decrease the adhesive curing time. The curing time of the adhesive was accelerated.

Kinloch[12] emphasized that the radical electrons, free ions and the working mechanism can make a surface region for polymer that need, to be cross-linked for having a surface treatment. The plasma can be processed by oxidation, polymerization or grafting.

Kraus *et al*.[13] studied low pressure plasma for polymer materials as PEEK and POM. They used the wetting method with an acid-base approach on polymer materials. Experimental studies showed that mechanical tensile strength values were raised with plasma treatment.

Moghadamzadeh *et al*.[14] studied different surface treatment techniques for WPC material before adhesive bonding. Flame, corona discharge treatment, mechanical abrasion and a combination with two different treatment techniques were used. Pull off strength was examined. Effective adhesive bonding was observed in the combination of both mechanical abrasion and corona discharge treatment techniques.

Han *et al*.[15] investigated the effect of carbon nanotubes (CNTs) on a PET surface. Prior to the surface modification of the CNTs, the desired area of the surface was established by multiple repetitions of the corona treatment applied on the PET material. Test results for adhesive strength between the CNTs and the PET material provided strong evidence that coronal surface treatment could be significantly increased.

Brewis *et al*.[16] studied organic molecules, including polymers that have low surface energies. Poor adhesion to some polymers is often tied to the polymer interior, but an alternative reason was suggested. The presence of weak boundary layers, that is, the bond region of low bond strength between the polymer and the polymer, is also a region to be considered.

Encinas *et al*.[17] studied three common polyolefin; high density polyethylene (HDPE), low density polyethylene (LDPE) and PP. These materials were surface treated with atmospheric pressure air plasma. After plasma treatment, different aging times were applied and contact angle measurements were made. The best aging times were between 21 and 31 days compared to the strength values obtained.

Encinas *et al*.[18] studied the effects of high strength bonding connections on surface preparation methods for polymeric materials (PP) and a PU based primer surface coating was investigated. The results showed that the plasma treatment strengthens the bonding area. The surface obtained by the abrasion prevented the uniform distribution of the bond on the entire surface.

Ku *et al*.[19] studied the plasma of PP on slip resistance between PP and aluminium plates by plasma treatment under atmospheric pressure. The optimum number of applications was determined by measuring contact angle, paint adhesion strength and changes in Scotch tape peel. Strength was measured as a function of the number of operations. The contact angle was shown to be influenced by atmospheric pressure.

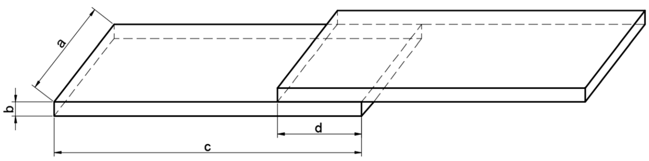
Yangrong *et al*.[20] investigated the effect of laser abrasion surface treatment on joint performance. Higher energy laser abrasion application on AA6022-T4 Al material showed a positive change in the water holding ability of the surface. Both the surface roughness and the energy of the surface area increased in the process with high energy flow.

Experimental

Material. Test specimens of 35×110 mm^2 were cut from a 5 mm thick sheet of PE, PP and PVC materials for shear stress tests. Test specimens of 25.4×44.5 mm^2 were cut from a 35 mm thick sheet and shaped for the izod impact test machine at the standard ASTM D950-3.[21] Technical drawings of shear stress test specimens for PE, PP and PVC are given in Figure 1. Izod impact test specimen is given in Figure 2.

Weicon Easy-Mix PE-PP 45 adhesive was applied on the adherend surfaces. It is a two component adhesive that is based on methyl acrylate with a processing time of approximately 2-3 min. It provides a fast development of strength, high final strength, curing with residual elasticity, ageing resistance and chemical resistance. It has average shear strength between 2.8 and 14.1 MPa on polymer surfaces and no test record on its impact strength.[22]

Surface Treatment. Three different surface treatment methods were applied to specimens before bonding. In the first step, three types of specimens were abraded with the same mesh size sandpaper (P100C). For the second treatment, three types of specimens were subjected to corona treatment for two different processing times. The first specimen group was subjected to corona treatment for approximately 150 sec, the second specimen group subjected to treatment for approximately 300 sec. For the third treatment, three types of specimens were subjected to plasma treatment for two different processing times, similarly to the corona treatment. After all surface treatment and bonding process were completed specimens were dried in an oven at approximately 50 ºC. Both corona and plasma treatments were applied according to



	Width	Thickness	Height	Overlap
	a*	b	c	d
PE	35	5	110	30
PP	35	5	110	30
PVC	35	5	110	15

*Units are milimetres (mm).

Figure 1. Shear stress test specimen for PE, PP and PVC.

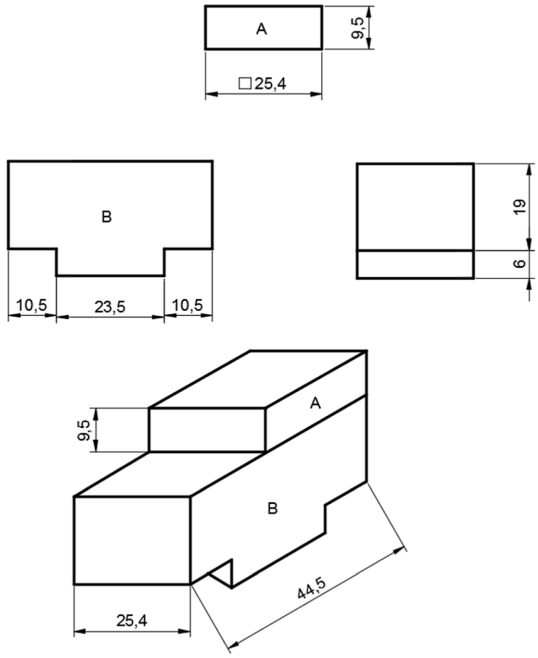


Figure 2. Izod impact test specimen.

Table 1. Technical Specification of Corona Treatment System[25]

Mains voltage and frequency	230/110 VAC, 50/60 Hz
Output	550 W
Output voltage	6.5 kV
Dimensions of operation heads	45-65 mm
Weight	13 kg
Timer	0.5-10 s

Table 2. Technical Specification of Plasma System[26]

Mains voltage and frequency	230 VAC, 50/60 Hz
Plasma power	Max 400 VP
Output voltage	300 W
Plasma treatment time	10-180 s
Plazma electrode system	Ceramic insulated
Dimensions (cm) Outer portion Inner portion	 60×41×34 12×18×5.5
Weight	36 kg

ASTM D6105-04[23] and ASTM D2093-03.[24] The technical specification of corona and plasma treatment systems is illustrated in Table 1 and Table 2.

Temperature Effect and Ageing. Temperature effects and ageing conditions were tested to show the effect of adhesive strength. Although the temperature effect is very serious for bonding, another major factor is moisture. In order to observe this situation, experimental studies were carried out at different temperature and humidity levels. The specimens to which applied surface treatments were incubated at four different temperatures (-20, 0, 20, 50 ºC) prior to shear testing. A deep freeze was used for -20 and 0 ºC. Ageing test temperatures, humidity level (95% Rh.) and ageing times (30, 60, 90 days) were determined according to standard EN 2243-5.[27] The cabinet working principle for temperature range is between -10 and 60 ºC and, humidity set range is between 10% and 95% Rh. The shear test was done at room temperature. Corona and plasma surface treatments were applied at two different levels which have 150 and 300 sec.

Bonding Process. The mechanical abrasion, corona and plasma processes were applied to the samples tested. Corona and plasma surface treatments were applied at two different levels. Weicon Easymix PE-PP 45 adhesive was applied on one substrate after the surface treatment. After bonding, the specimens were carefully protected. The specimens were left for 24 h at room temperature to complete curing. After surface treatments and bonding, the specimens used for shear stress tests were left 30 days in the deep freeze at -20, 0 ºC and the other specimens were exposed to 50 ºC and 95% Rh. in the climatic cabinet for 30, 60 and 90 days for ageing tests.

The bonded specimens were loaded in shear using an Alsa servo-hydraulic testing machine at across head displacement rate of nearly 2 mm.min^{-1}. In experiments, an average of four specimens in each group was obtained. The experimental conditions of the shear tests are summarized in Table 3. Measuring for impact strength, the specimens which had been used for izod impact tests were left for 30 days in deep freeze at -20 and 0 ºC. Corona and plasma surface treatments were applied in one level. The experimental conditions for the izod impact tests are summarized in Table 4.

Table 3. Experimental Conditions for Shear Stress Test

Material	Surface treatment	Test conditions*					
		-20 ºC	0 ºC	20 ºC	50 ºC±3 ºC, 95-100% Rh		
					30 days	60 days	90 days
PE	Untreated	1.1	1.8	1.6	-	-	-
	Abraded	1.1	1.2	1.7	2.0	1.2	1.6
	Corona 1st level	3.7	2.8	3.4	3.7	1.4	0.8
	Corona 2nd level	3.9	2.3	2.6	3.4	1.5	2.0
	Plasma 1st level	3.8	2.7	2.6	3.7	3.1	1.9
	Plasma 2nd level	2.8	2.4	2.9	3.5	2.3	1.8
PP	Untreated	1.6	1.1	1.2	-	-	-
	Abraded	1.8	3.2	2.2	2.8	2.0	2.2
	Corona 1st level	5.4	4.9	4.5	3.3	2.6	2.2
	Corona 2nd level	4.2	4.8	3.2	3.3	1.4	2.1
	Plasma 1st level	4.8	3.9	5.5	4.6	3.3	2.1
	Plasma 2nd level	5.4	2.6	3.5	3.5	2.3	1.0
PVC	Untreated	2.0	1.4	1.9	-	-	-
	Abraded	11.0	9.8	10.9	10.3	8.8	4.5
	Corona 1st level	8.1	9.3	8.0	6.7	8.1	2.0
	Corona 2nd level	5.6	6.5	6.9	5.7	4.5	4.5
	Plasma 1st level	11.5	9.4	10.9	3.9	3.9	4.0
	Plasma 2nd level	8.1	7.5	7.6	4.7	4.5	3.7

*Mean values of shear stress for four specimens (MPa).

Table 4. Summarization of Experimental Conditions for Izod Impact Test

Material	Surface treatment	Test conditions*		
		-20 ℃	0 ℃	20 ℃
PE	Untreated	2.1	3.6	1.9
	Abraded	6.0	5.0	6.2
	Corona 1st level	3.6	4.2	4.2
	Plasma 1st level	4.0	3.6	5.2
PP	Untreated	2.1	2.1	1.9
	Abraded	6.5	6.4	6.4
	Corona 1st level	0.8	3.8	3.0
	Plasma 1st level	2.9	4.3	4.9
PVC	Untreated	1.9	3.1	4.2
	Abraded	9.3	11.2	11.0
	Corona 1st level	4.7	6.7	6.7
	Plasma 1st level	6.6	5.8	7.2

*Mean values of impact strength four specimens (kJ/m^2).

Results and Discussion

The experimental studies were analysed. The results were interpreted by comparison. PE, PP and PVC material specimens were evaluated individually according to the process applied. Figure 3 shows the shear stress changes due to different temperature and surface treatments which were applied for PE specimens. The results showed that the conditions of -20 ℃ in deep freeze for 30 days and 50 ℃ in cabinet for 30 days' ageing for which had corona treatment had the highest results for shear stress. In the same conditions, the plasma treatment specimens provided similar results. As the ageing time increased, the shear stress was reduced. In general the ageing time increases at the same temperature and, the shear stress falls.

Figure 4 shows the shear stress changes due to different temperature and surface treatments applied to PP specimens. The highest values were shown at -20 ℃ in deep freeze for 30 days and at room temperature for 30 days. First level corona and first level plasma provided nearly the same values. Generally, ageing time decreased the shear stress for the effect of treatment on every type of surface.

Figure 5 illustrates the shear stress changes due to different temperature and surface treatments applied to the PVC specimens. PVC shows the highest effect of surface treatment at -20 ℃ in deep freeze for 30 days and at room temperature for 30 days. As ageing time increased, shear stress decreased, as for PP.

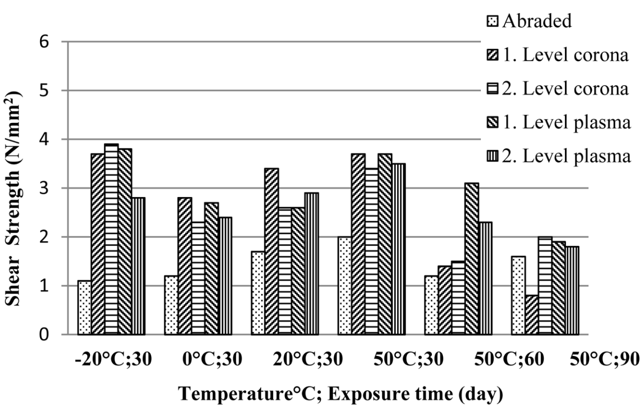


Figure 3. Shear strength of joints as a function of temperature and applied surface treatment for PE.

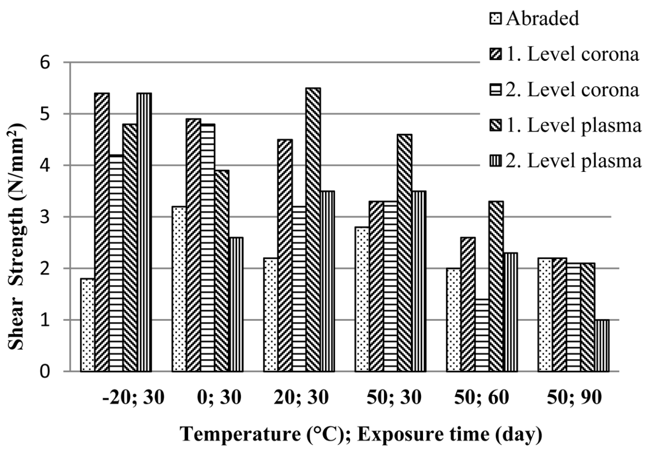


Figure 4. Shear strength of joints as a function of temperature and applied surface treatment for PP.

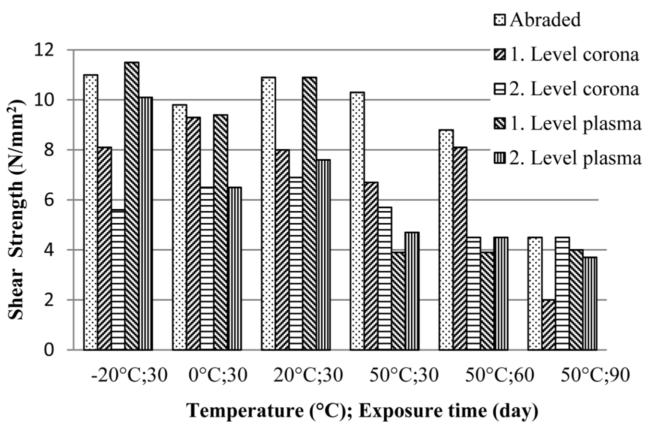


Figure 5. Shear strength of joints as a function of temperature and applied surface treatment for PVC.

The effects of surface treatment showed with abrasion, and with corona and plasma surface treatments. The abrasion of PE, PP and PVC specimen's shear stress changes with temperature and exposure time are shown in Figure 6. The abraded surfaces for PE and PP provided similar results but PVC had a characteristic difference because of its friability. The results for PVC began to decrease with increasing of temperature and ageing time. According to PVC results, ageing temperature and ageing time increases showed the failure because of the decline of the interface's bond strength. Although PVC worked more at a temperature of 50 °C, the adherend and adhesive interfaces could not provide shear strength for all the shear stress values for PE specimens with abrasion and this is shown in Figure 6. Shear stress values of abraded specimens decreased with the effect of ageing. The PVC specimen in particular was shown to have a net decrease in prolonged ageing time.

The shear stress values for specimens to which first level corona was applied are shown in Figure 7. PE and PP specimens showed better results for shear stress values than abrasion treatment. Corona treatment were able to give better results for shear stress until beginning of ageing. Prolonging ageing time gave the worst values for shear stress in all the specimens.

For second level corona treatment, the best values for PVC were shown at room temperature. This is shown in Figure 8. Temperature increases weaken van der Waals bonds. At the room temperature, shear stress values are high but the elongation is low.

For the first level plasma, PE and PP specimens decreased when ageing started. This is shown in Figure 9. The ageing time becomes longer, the shear stress values are lowered. PVC in particular showed a different tendency than PE and PP. The temperature rise led to a decline in shear stress values for PVC specimens. PVC surfaces to which plasma treatment was applied lost the effect of ambience as with temperature increases. Surface energy had a high value at low temperatures.

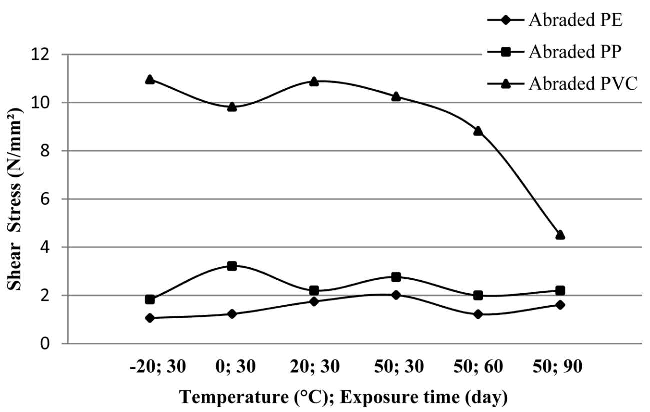


Figure 6. Shear stress of abraded PE, PP and PVC as a function of temperature.

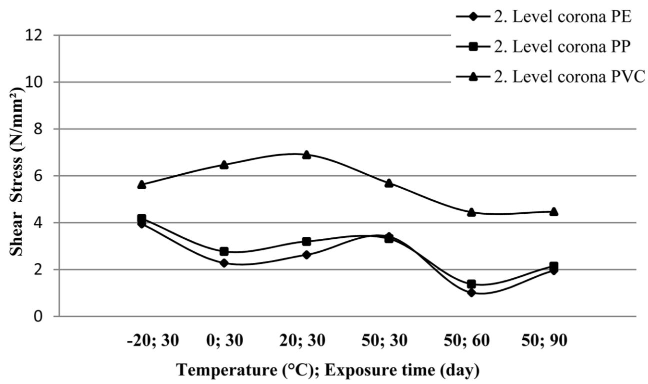


Figure 8. Shear stress of second level corona PE, PP and PVC as a function of temperature.

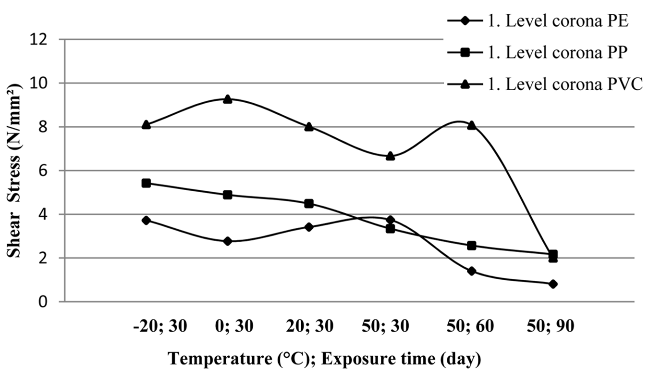


Figure 7. Shear stress of first level corona PE, PP and PVC as a function of temperature.

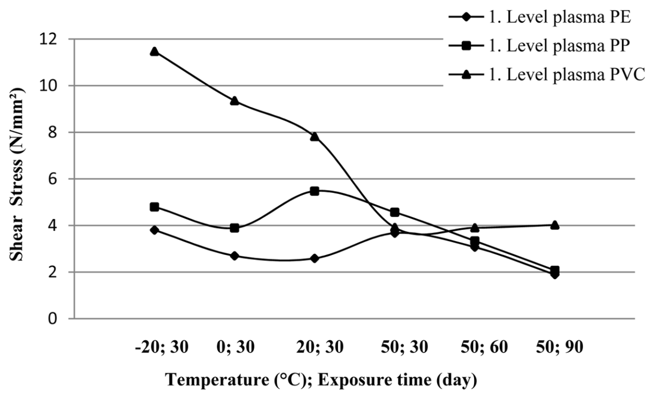


Figure 9. Shear stress of first level plasma PE, PP and PVC as a function of temperature.

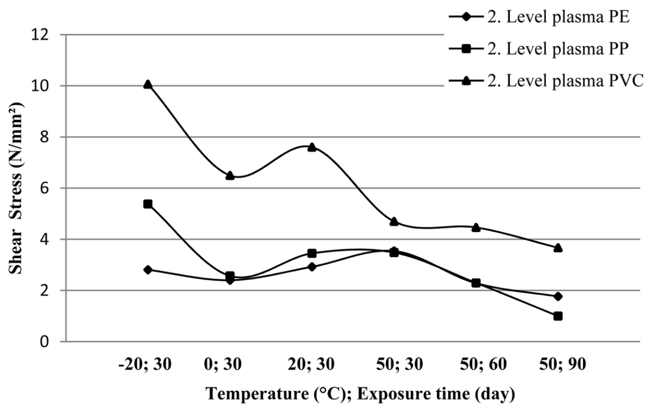


Figure 10. Shear stress of second level plasma PE, PP and PVC as a function of temperature.

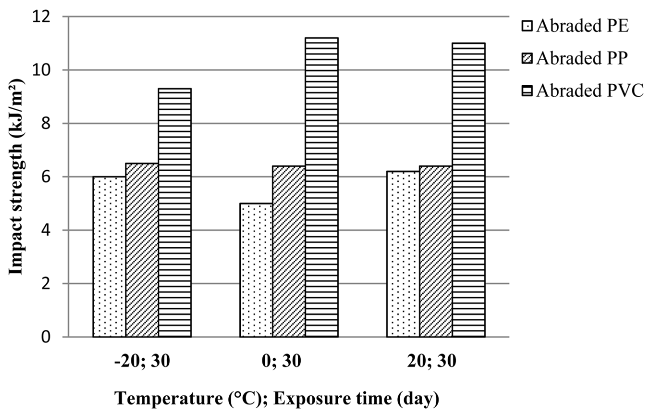


Figure 11. Impact strength of abraded PE, PP and PVC as a function of temperature.

For the second level plasma PE had the highest value of shear stress at 50 ºC; 30 days of ageing. PP had the highest value at -20 ºC; 30 days in deep freeze. PVC had two different highest values between -20 and 20 ºC after 30 days in deep freeze. This is shown in Figure 10. It shows that the variation in the surface of the resulting bond interface in which shear stress change depending on the temperature and gives different results depending upon the surface treatment. Specimens were incubated at three different temperatures for 30 days which used on impact tests. Figure 11 shows the impact strength values for abraded PE, PP and PVC as a function of temperature.

For the results obtained by abrasion treatments, the highest value belonged to PVC. PE and PP showed nearly similar values for impact tests. Impact strength was obtained at 20 ºC temperature and this was higher for first level corona treatment (Figure 12).

The highest strength value was obtained in PVC specimens. PP specimens showed better values as temperature increased.

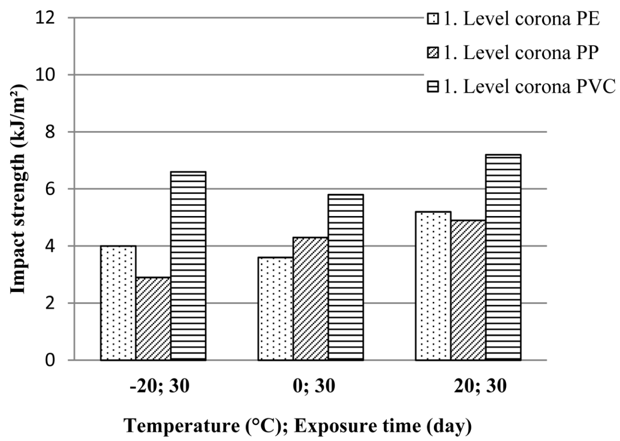


Figure 12. Impact strength of first level corona PE, PP and PVC as a function of temperature.

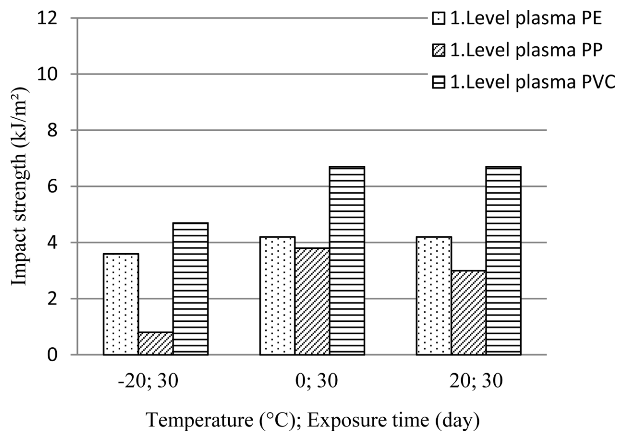


Figure 13. Impact strength of first level plasma PE, PP and PVC as a function of temperature.

At first level plasma treatment, the highest impact strength values were obtained at 0 ºC (Figure 13).

Conclusions

In this experimental study, effect of surface pre-treatment and temperature changes were investigated the adhesively bonded polymer joints. PE specimens had the best results for shear strength on the -20 ºC; 30 days and 50 ºC; 30 days ageing conditions which has applied the corona and plasma surface treatments. On different temperature and time, the adhesion bond has been obtained successful results on interface for resistance on -20 ºC and aging at 50 ºC.

The longer the time taken for ageing for adhesion and aged samples, the more movement starting from intermediates led to a weakening of the joints. Glass transition temperatures are -115,

-20, 80 ºC for PE, PP and PVC, respectively. So, the shear strength of PVC at an operating temperature of -20 ºC is higher than other materials. As the glass transition temperature of the material moves away from the operating ambient temperature, the strength value increases. The decrease in strength of PP is because its operating temperature is close to the glass transition temperature.

PP specimens gave the most successful result for shear strength at -20 ºC; 30 days after corona treatment has been applied. The interface bonds are weakened while the temperature has increased. When the PP material approaches the glass transition temperature, there is breakage in the bond structure. Therefore, weak adhesion forces occurred between the polymer and adhesive interfaces. The joint strength has been increased by the corona system. The effect of temperature on plasma treated surfaces is minimal. Plasma surface treatments carried out at room temperature on the PP specimen is determined to have a higher shear strength.

The surface treatments which were applied to PVC made improvements to the adhesion forces. So, corona and plasma surface treatments gave higher joint shear strength. Although the application of surface treatment to PVC gave the most successful results for the impact strength, PE and PP did not. The specimens to which abrasion treatment had a higher impact strength than corona and plasma treatment.

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