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EXPERIMENTAL STUDY OF TEMPERATURE EFFECT AND CURING TIME ON THE SHEAR STRENGTH OF ADHESIVE JOINTS BY POLYVINYL PYRROLIDONE PVP K30

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ABSTRACT

This research represents an experimental work to study the effect of temperature and curing time on the shear strength of adhesive joints by polyvinyl pyrrolidone PVP k30. In this work, shear strength of the adhesive made from dissolving 8 grams of polyvinyl pyrrolidone (k30) in 50 ml of ethanol alcohol high purity 99.9% was studied. This study focused on the effect of temperature and curing time under pressure of a fixed rate of 5 MPa. Therefore, the effect of five temperatures (200, 215, 225, 235 and 250) °C for different times (10, 20 and 30) minutes under the influence of the same pressure in all cases was studied by taking pictures of samples using Scanning Electron Microscope (SEM). It was inferred that the best resistance obtained at a temperature of 225 °C and time of 10 minutes, reaching overhead hanging to 1669 N, that there is an increase in the resistance of the adhesive with the increase in temperature and up to 225 °C, and then there is a decrease in the resistance and up to 250 °C at the time of 10 minutes.

KEYWORDS: Adhesive, Shear strength, Temperature, Poly vinyl pyrrolidone, curing time.

دراسة عملية لتأثير درجة الحرارة ووقت المعالجة على مقاومة القص للوصلات الملصوقة بواسطة البولي فينيل بروليدون PVP k30

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الخلاصة

يمثل هذا البحث عمل تجريبي لدراسة تأثير درجة الحرارة وزمن المعالجة على مقاومة القص الملتصقة بمادة PVP K30 . في هذا العمل تمت دراسة مقاومة القص للمادة اللاصقة المصنوعة من اذابة 8 غرام من البولي فينيل بايرولدين K30 في 50 ملليتر من كحول الايثانول عالي النقاوة 99.9 %. ركزت هذه الدراسة على تأثير درجات الحرارة وزمن التسخين على مقاومة المادة اللاصقة و تحت ضغط ثابت مقداره 5 ميجا باسكال وتم دراسة تأثير خمس درجات حرارة وهي (200, 215, 225, 235, 250) درجة مئوية لأزمنة مختلفة هي 10 و20 و30 دقيقة تحت تأثير نفس الضغط في جميع الحالات كما تم التقاط صور لعينات مجهرية بواسطة المجهر الماسح الالكتروني ، تم استنتاج ان افضل مقاومة حصلت عند درجة حرارة 225 مئوية ووقت 10 دقيقة حيث وصل مقدار الحمل المسلط الى 1669 نيوتن، ان هناك زيادة في مقاومة المادة اللاصقة مع زيادة درجة الحرارة ولغاية 225 درجة مئوية ومن ثم هناك نقصان في المقاومة ولغاية 250 درجة مئوية عند زمن 10 دقيقة.

1. INTRODUCTION

Successful bonding of parts requires an appropriate process. The adhesive must not only be applied to the surfaces of the adherents, but the bond should also be subjected to the proper temperature, pressure, and hold time (Ebnesajjad and Landrock, 2007). Starch-based adhesives were developed by hydrolyzing starch with PVA under alkaline and acidic conditions at various treatment temperatures. The results showed that the joint strength of starch adhesives reached a maximum value at 40 °C. When the treatment temperature was 55 °C, the crystalline of treated starch was the lowest and the thermal resistance also the weakest; it decreased by 10.1% and 13.6%, respectively compared with untreated starch. Obvious erosion could be observed from the SEM images of treated starch (Yu et al., 2015). The temperature affects on the behavior of two adhesive systems (acrylic and epoxy) and on the capacities of adhesively bonded lap shear joints. The temperature influence is quantitatively described for short-term loading over a service range of temperature from -20 °C to +40 °C. The quantitative description is done by proposing the partial factors and the conversion factors that take the temperature effect into account. This influence is also dealt with for long-term loading to describe the shear creep behavior of the adhesive materials used. Consequently, the time-to-failure of the bonded lap shear joints due to the creep phenomenon of the adhesives under three applied stresses at room temperature is predicted. Moreover, the estimation of time-to-failure is extended to be used for other shear stress levels. The temperature influence as well as the efficiency of using adhesive-bonded joints in lightweight galvanized steel constructions is also illustrated by giving a practical example of strengthening cold-formed "C" section girders. Comparisons between the two adhesive systems for all cases are given (Sahllie, 2015).

The combined effect of the temperature and test speed on the tensile properties of a high temperature epoxy adhesive was investigated. Tensile tests were performed at three different test speeds and various temperatures (room temperature (RT) and high temperatures (100ºC, 125ºC and 150ºC)). The glass transition temperature (Tg) of the epoxy adhesive investigated is approximately 155ºC. The ultimate tensile stress linearly decreased with temperature (T) while logarithmically increased with the loading rate, which is in the accordance with the Airing's molecular activation model (Doina, 2011). The test of mechanical properties of as list of adhesives was conducted at different temperatures from room temp. to 100 K. This material was candidated to use in spaceflight hardware assembly in one of NASA programs, the material properties which measured were Young's modulus, Poisson's ratio, and the coefficient of thermal expansion. The aim of the work was to improve the shear strength of adhesive joints by using best value of temperature and heating time at certain pressure (Cease et al., 2006). The cohesive–zone model approach was used to study fracture mechanics in lap-shear joints, the authors investigated the limit of using linear-elastic fracture mechanics. Experimental and numerical study were done on a joint system of aluminum and commercial adhesive, using finite element solve with ABAQU5.8 code software. By employing data of the maximum traction–separation law, most researchers studied the adhesive joint fracture behavior by linear elastic fracture mechanics when the substrates remain elastic, and when the adherents deformed plastically, they gave a focus on transition for the zone between the elastic and plastic regions (Kafkalidis and Thouless, 2002). The motive behind the present work is to produce an adhesive material for metals and is characterized by strong high-cut and bearing high temperatures up to 250^{o}C as the wholesale production is cheap compared to other types of adhesives.

2. EXPERIMENTAL WORK

2.1. Materials

Polyvinyl pyrrolidone (PVP. K30) has linear formula $[C_6H_9NO]_n$ which a molecular weight M.W 40,000 was used in this work as glued material with a molecular weight 40000 gm.mol^{-1}. It has viscosity 26 cps and was tested by using viscosity measurement device for 0.004 mol./L concentration. The melting point of PVPk30 is 172^{o}C. The adhesive material was prepared of concentration of 0.004 mol. L^{-1} (PVP-K30) by solving 8 gm. of PVP powder in 50 ml of high purity of ethanol 99.9% placed on stirrer device for 30 minutes at room temperature according to equations (1) & (2) :

$$C_x = \frac{n_x}{V} \tag{1}$$

$$n_x = \frac{g}{M.W} \tag{2}$$

Where : C_x is the concentration mol. L^{-1} , and n_x is the number of moles.

V = 50 ml volume of solvent , g = 8 gm. is the mass of PVP

M.W = 40000 gm. mol^{-1} is the molecular weight.

After substitute the above quantities then C_x = 0.004 mol. L^{-1}

2.2. Preparation of adhesive joint

The single shear lap joints of adherent were Copper specimens with dimension (25.4 mm * 1.6 mm * 90 mm) having a contact area 330 mm^2 of the adhasive joint that was prepared according to ASTM D1002 (ASTM, 2001), as shown in the Fig. 1.

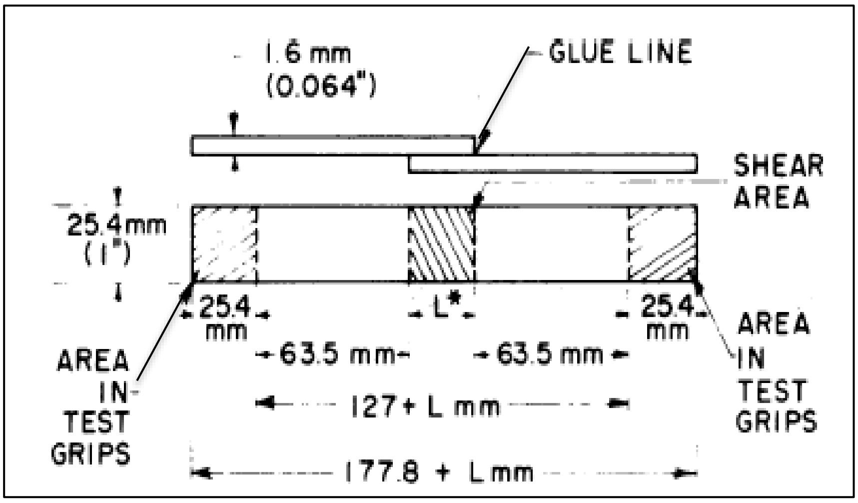

Fig. 1. Adhesive lap joint specimen according ASTM D1002 -01.

The copper specimens were pressed under pressure 5 MPa and heating temperatures (200, 215, 225, 235, and 250) °C for various times (10, 20 and 30) minutes by using press heating system, as shown in the Fig. 2. Fig. 3 show the pressed copper adherents.

Pressure 5 MPa was selected depending on some references and conducting some laboratory tests. First, these tests were carried out at pressure less than 5MPa, but it was found that the adhesion of metallic specimens did not occur when adding adhesive material under this pressure at the same conditions, i.e. at temperature range 200 - 250 °C. Consequently, other test were then performed at pressure higher than 5MPa, and it was found the adhesion took place without adding the adhesive material due to pressure increase at temperature range 200-250°C.

The temperature range 200-250°C was selected to be in consistent with the adhesive material properties. Where, the melting point of PVP is 172°C according to the melting temperature test, while the decomposition temperature reach to 483°C according to TGA test, so the required temperature is with in this range.

Curing times (10,20,30) minutes were selected to compare among them and since increasing the curing time in a test for more than 30 minutes caused burning of the adhesive material, formation of a black adhered region, the existence of cracks in the adhesive material structure and weakness in shear strength.

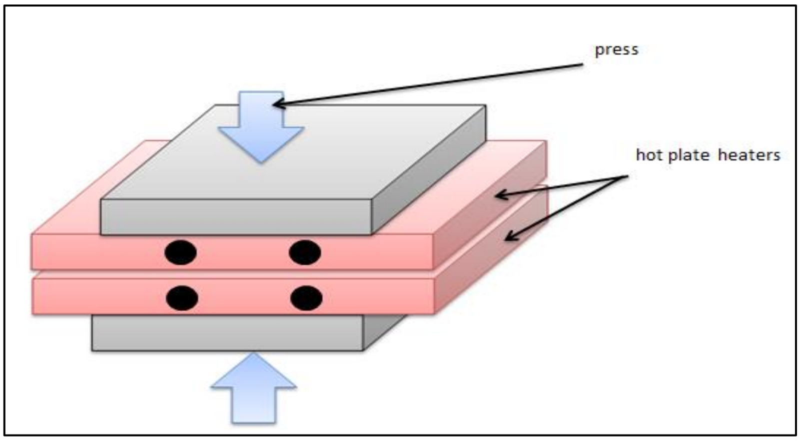

Fig. 2. Schematic of press heating system.

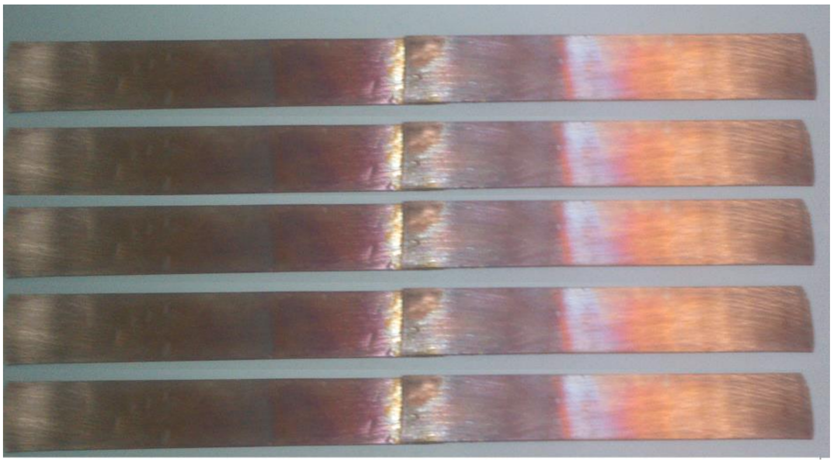

Fig. 3. Copper adherents after pressing.

2.3. Shear test

The shear strength of the joint was measured by using a universal tensile testing machine by pulling apart the two plates with a rate of 1 mm/min^{-1}, as it can be seen on the scheme presented in Fig. 4.

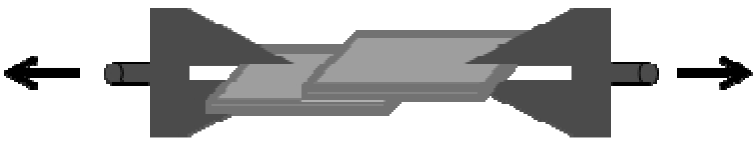

Fig. 4. Shear test.

3. RESULTS AND DISCUSSIONS

Fig. 5 shows the Fourier transform infrared spectroscopy (FTIR) spectrum of the PVP polymer powder in the range (4000-400) cm^{-1}. A broad peak located at 800 cm^{-1} is due to the outer face vibration oscillation of the hydroxyl group (O-H), and the strong peaks centered at 1530 cm^{-1} and 1320 cm^{-1} are assigned to the inner face bending vibrations of the hydroxyl group. Also, a broad band at 2953 cm^{-1} has been observed in the spectrum of PVP, and the vibration band of C=O group appears at 1669 cm^{-1} suggesting some H-bonding carbonyl groups exist in PVP. A broad and intense band in the range 3464~1669 cm^{-1} is ascribed to the stretching vibration of the associated hydroxyl group. The bands at 2953 and 1669 cm^{-1} are attributed to the characteristic stretching vibrations of C-H, band respectively. These results approximate to which in (Sivaiah et al., 2010).

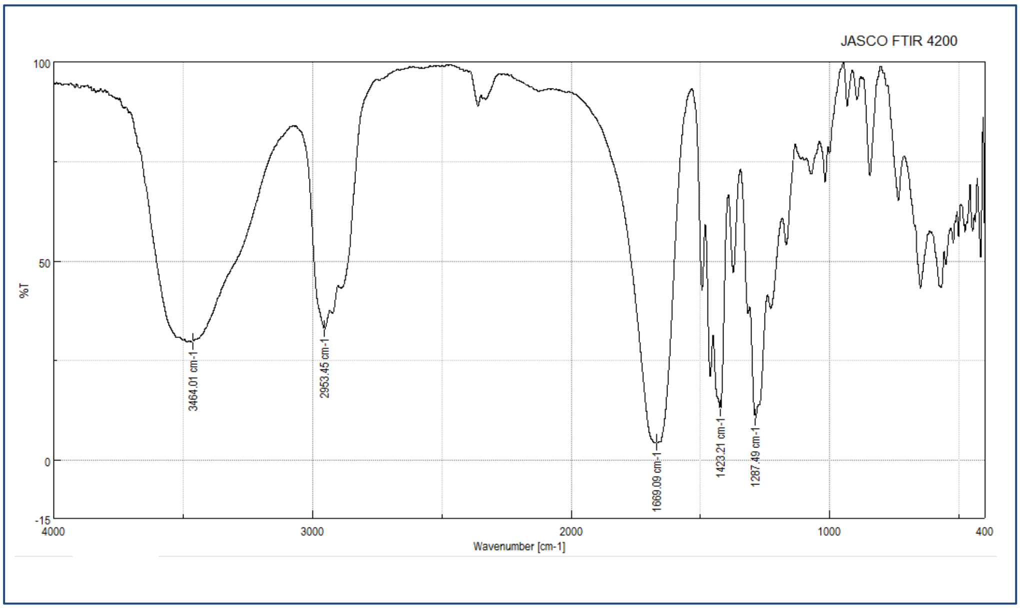

Fig. 5. FTIR profiles of PVP k-30.

Fig. 6 depicts the TGA-DTA profiles of PVP polymer. The Thermo Gram Analysis (TGA) has been plotted with weight loss as a function of the temperature for the reference PVP precursor with a heating rate of 10 °C/min in the temperature range from 40 to 800 °C. It is clear that the initial weight loss from the TGA curve is 12% in the temperature range 40-87.9 °C, this is due to loss of OH content. In the Differential Thermal Analysis (DTA) curve, two exothermic peaks were observed at 483.92 °C and 678.08 °C, respectively. The sharp and strong exothermic peak at 483.92°C is due to the combined effect of combustion of organic residuals and the decomposition of PVP and which is well above the heating temperature employed in the present work .These results approximate to which in (Sivaiah et al., 2010).

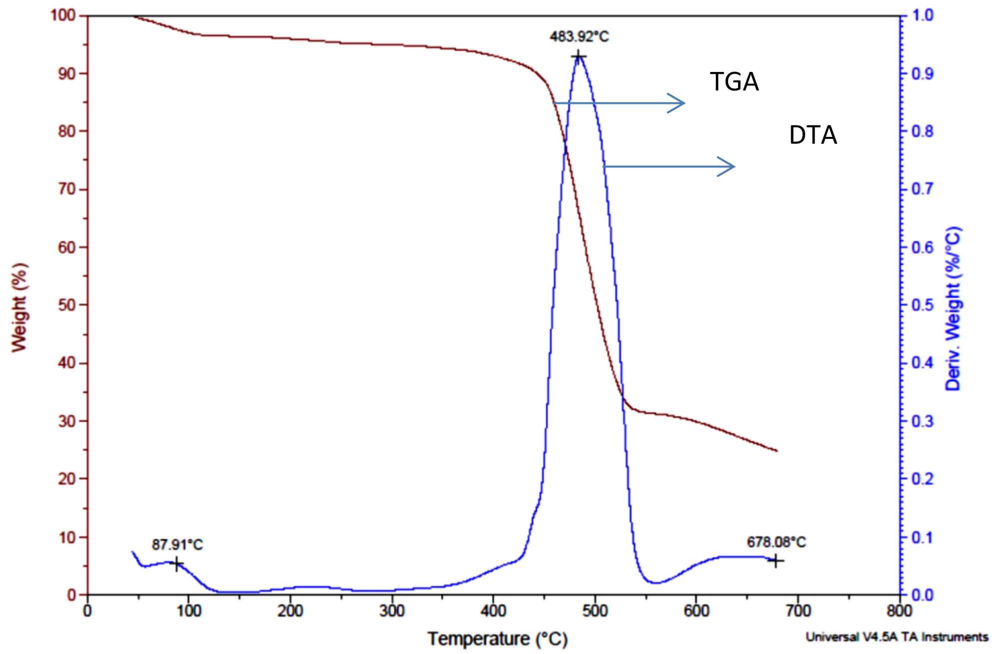

Fig. 6. TGA-DTA profiles of PVP k-30.

Fig.7 shows the relationship between curing temperature and load for 0.004 PVP at 10 minutes under 5 MPa. There is a slight proportion increment (2.2%) from (200 to 225) ^{o}C reaching to the best which is 225^{o}C with load is 1669 N while the relationship is inverse proportion from (225 to 250) ^{o}C because the strength of adhesive joint depends on the viscosity of the adhesive, which depends mainly on temperature; increasing the temperature lowers the viscosity and in turn reduces load.

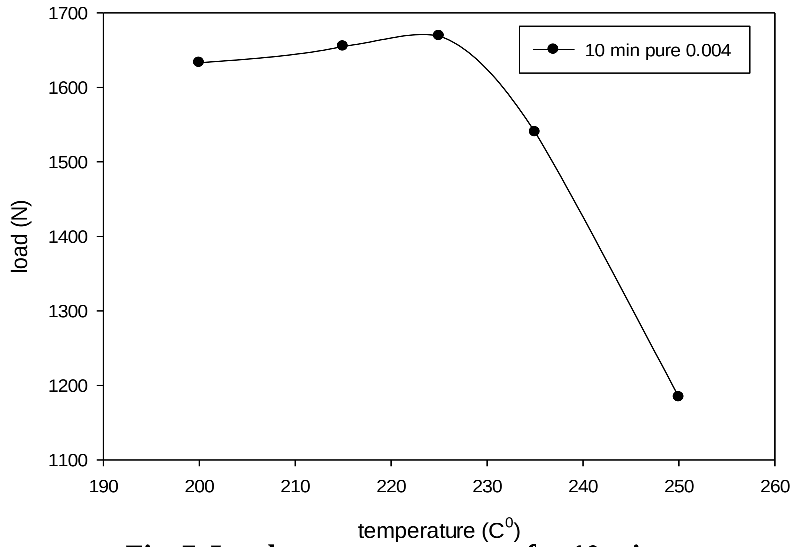

Fig. 7. Load-temperature curve for 10 min.

Fig. 8 reveals the relationship between curing temperature and load for 0.004 PVP at 20 minute under 5 MPa. There is a direct proportion increase (13%) from (200 to 225) ^{o}C reaching to the best which is 225 ^{o}C with load 1430 N, while the relationship is inverse proportion from (225 to 250) ^{o}C because the strength of adhesive joint depends on the viscosity of the adhesive, which depends mainly on temperature; increasing the temperature lowers the viscosity and in turn reduce the load.

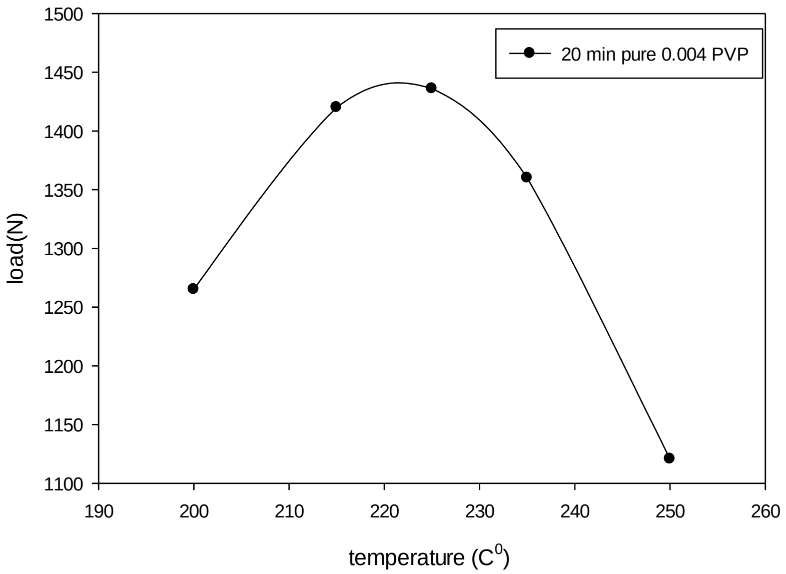

Fig. 8. Load– temperature curve for 20 min.

Fig. 9 shows the relationship between curing temperature and load for 0.004 PVP at 30 minute under 5 MPa. There is inverse proportion from (200 to 250) ^{o}C, and the best is 200 ^{o}C with load 1517 N because of the strength of adhesive joint depends on the viscosity of the adhesive, which depends mainly on temperature; increasing the temperature lowers the viscosity and in turn reduces the load.

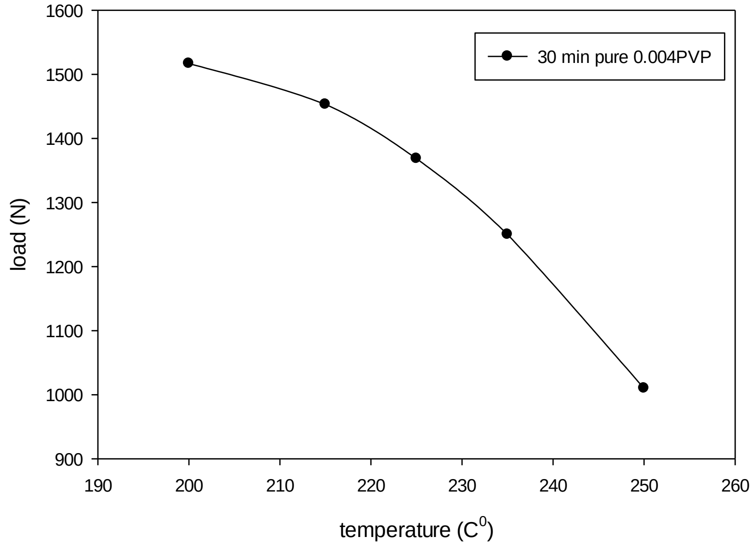

Fig. 9. load– temperature curve for 30 min.

Fig.10 demonstrates the comparison relations between curing temperature and load for 0.004 PVP during the whole time range (10-30) minutes under 5 MPa, and the best is 225 °C of 10 minutes with load 1669 N because the strength of adhesive depends on the viscosity of the adhesive, which depends mainly on temperature; increasing the temperature lowers the viscosity and in turn reduces the load.

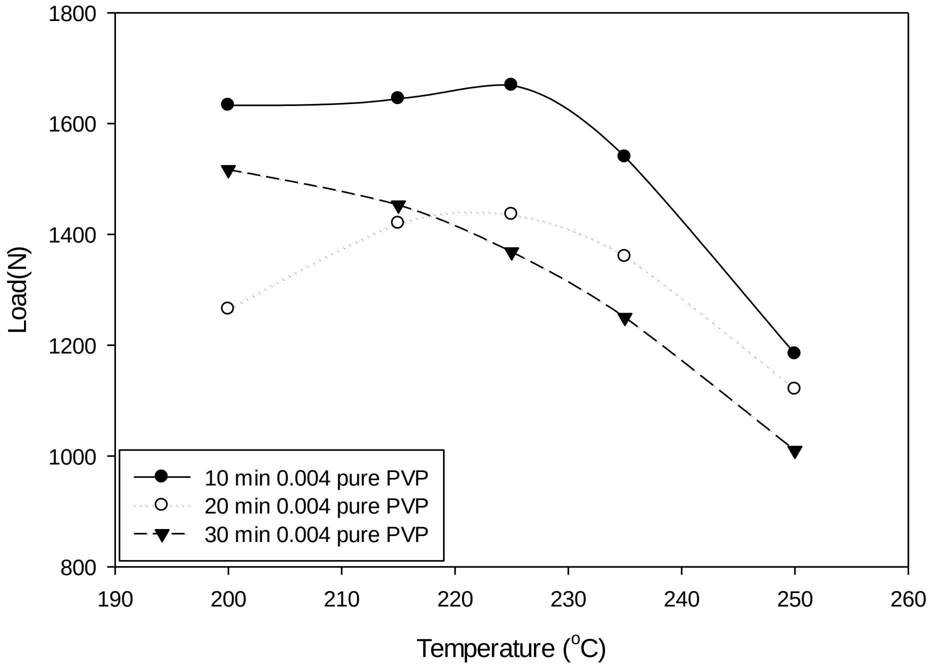

Fig. 10. Load-temperature comparison relations.

Table 1 shows the summary of results of curing temperature, load and heating time. There are three glued adherents for each temperature of each time for 0.004 PVP K30 at pressure 5 MPa.

This table manifests that the increasing in temperature and heating time causes a decrease in shear load because there is losing in viscosity of glued material. This result is comparable with (AL-Shammari et al., 2011).

Table 1. Shear strength for pure 0.004 PVP.

Curing time (min)	Temperature (^oC)	Shear load (N)	Shear strength (MPa)
10	200	1633	4.95
	215	1645	4.98
	225	1669	5.05
	235	1540	4.66
	250	1184	3.59
20	200	1265	3.83
	215	1420	4.30
	225	1430	4.33
	235	1360	4.12
	250	1121	3.39
30	200	1517	4.59
	215	1453	4.40
	225	1368	4.14
	235	1250	3.79
	250	1010	3.06

Fig. 11 exhibits the scanning electron microscope (SEM) images of adherents surfaces after shear test for each temperature and heating time. This picture shows that temperature 200 ^oC and a time of 10 minutes were enough to obtain the cohesion of the adhesive between the surfaces. This conclusion expresses the low value of load 1633 N inTtable1 and Fig. 7.

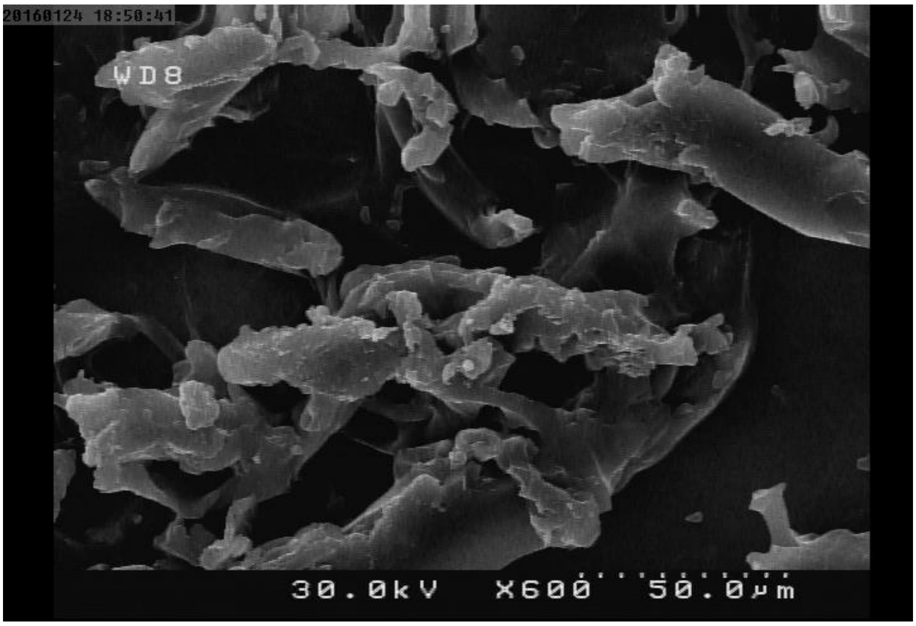

Fig. 11. SEM image for 10 min at 200 ºC.

Fig. 12 shows the scanning electron microscope (SEM) images of adherents surfaces after shear test for each temperature and heating time. This picture depicts that the best load of adherents accrued at temperature 225ºC and curing time 10 minutes because of the homogeneity of the distribution of adhesive on the surface, this leads to best value of load 1669 N in Table 1 and Fig. 8.

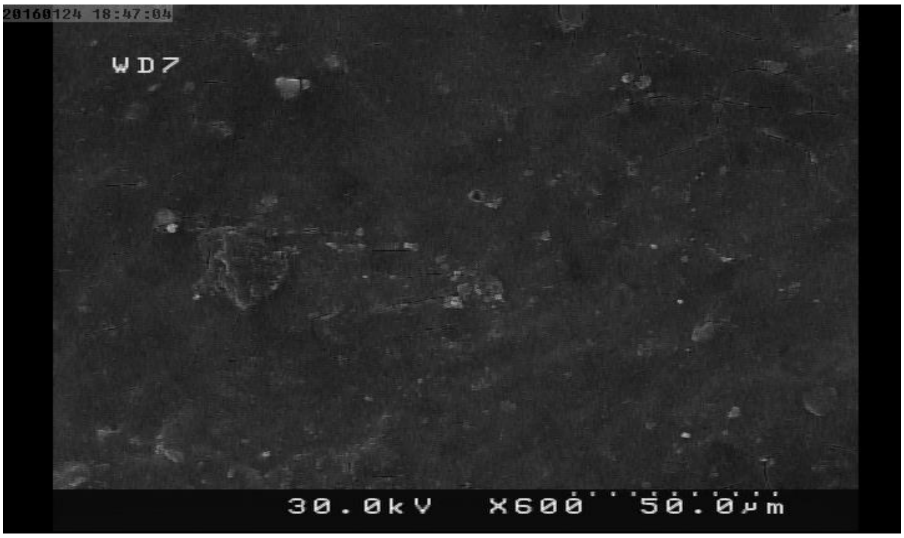

Fig. 12. SEM image for 10 min at 225ºC.

Fig. 13 displays the Scanning Electron Microscope (SEM) images of adherents surfaces after shear test for each temperature and heating time. This picture shows that cracks appear in adhesive layer, because there is a loss in the viscosity of adhesive due to heating to 250 ºC. This reason explains the low value of load (1184 N) in Table 1and Fig. 9.

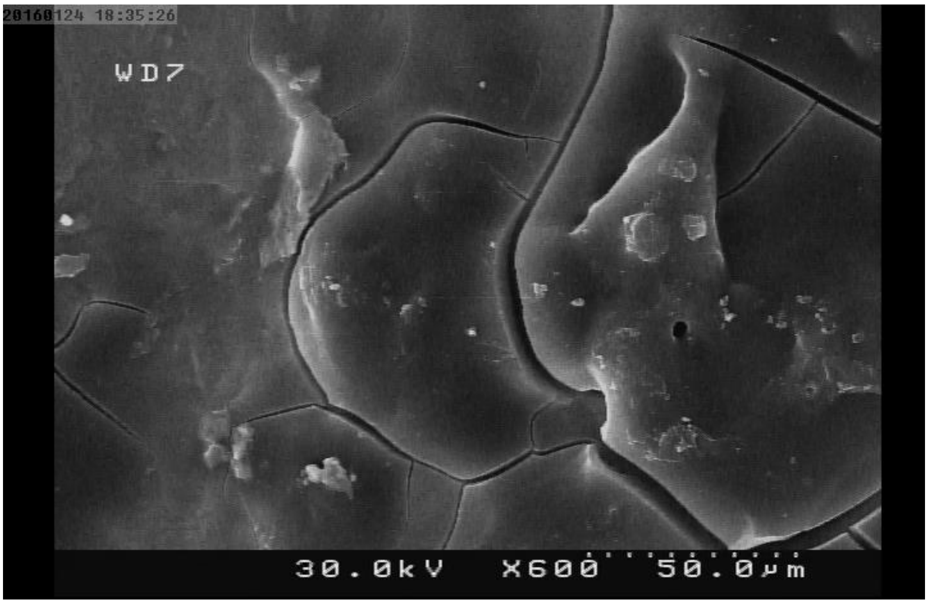

Fig. 13. SEM image for 10 min at 250^{o}C.

4. CONCLUSIONS

From the above results, the following conclusions can be written:

1. The temperature effect on the shear strength is a slight direct proportion from (200 to 225) ^{o}C but it is inverse proportion from (225 to 250) ^{o}C.
2. Increasing in heating time caused weakness in shear strength of lap joints .
3. The best value of adhesive joint loading is for 10 min and 225^{o}C because the viscosity of PVP k30 increased slightly in direct proportionality from (200-225) ^{o}C but decreased during the range (225-250) ^{o}C.
4. There is non-homogeneity in the microstructure of adhesive joints at temperature 200 ^{o}C, but there is homogeneity at temperature 225 ^{o}C
5. There is a degradation of shear strength of lap joints at temperature 250 ^{o}C due to the crack formation in the microstructure of adhesive joints.

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