Effects of various particles size of PKM as filler in Melamine-Urea-Formaldehyde (MUF) resin on performance.

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ABSTRACT

Palm kernel meal (PKM) which is the by-products of palm oil industry consists of considerable amount of protein that showed its great potential to enhance shear strength performance of MUF resin. Besides, due to the high price of melamine, it is attempted to reduce the content of melamine in MUF resins to as low as possible. So, in this study, PKM was used as filler in MUF resin to study its effects toward the shear strength performance. In practical, the shear strength performance tests are done by using automated bonding evaluation system (ABES). However, ABES is a destructive test that only produces one point of data per test. So, in order to make the evaluation of the prediction data more convenience, a response surface methodology D-optimal method was approached. From the results, it has shown that increasing in temperature and pressing time is not always true in obtaining higher shear strength performance for MUF resin with PKM as filler. Smaller the particle size of PKM filler in MUF resin, shorter time the high shear strength performance can be achieved. Also, the relationships of temperature, pressing time and particle size of PKM to obtain optimum and maximum shear strength were studied.

Keywords: PKM; MUF resin; particle size; filler; shear strength

INTRODUCTION

From the statistical data has shown that in Malaysia domestic crude oil output is expected to increase only by about 2 percent to 17.9 MMT. With an expected exportable surplus of about 15.0 MMT of palm oil and 1.0 MMT of palm kernel oil in MY2008/09, Malaysia will remain a formidable competitor in the world vegetable oil (U.S. Embassy 2009).

In line with the big increase in palm kernel crush, palm kernel meal (PKM) production is expected to increase by 2.1 percent to 2.4 MMT in 2008/2009. PKM is essentially a by-product of the palm oil industry, it is used primarily in cattle feed.With a very small domestic beef and dairy cattle sector, only minimal quantities are consumed locally. 2.05 MMT of PKM was exported with the bulk going to the Netherlands, New Zealand, Germany and South Korea. Due to the banning on the use of meat and bone meal in various countries has opened many overseas markets for Malaysian PKM exports. With an expected small increase in palm kernel crush in 2009/10, 2.1 MMT of PKM should be available for exports (U.S. Embassy 2009).

Hence, due to the abundant availability of PKM in Malaysia, PKM should be optimized for its usage. As from the research analysis, PKM consist of about 14-21 percent of protein after solvent extraction process (Boateng et al. 2008). This considerable amount of protein content in PKM has shown the potential in making wood adhesives. One of the major factors that contributed for the cross-linkage formation in wood adhesive bonding is the amino-groups ($-NH_2$). And thus, the availability of amino groups in protein content of PKM has shown great potential for wood adhesive industries.

Classical commercial MUF resins usually synthesized with high level of melamine (>50%) (No B. Y. et al., 2004). Due to the high price of melamine, it is

attempted to reduce the content of melamine in MUF resins to as low as possible depending on the purposes of use (No B. Y. et al., 2004 and Angelatos A. S., 2004). As a result, with the past history of these resins having focused on the effort to decrease the weight proportion of melamine in relation to the proportion of urea in the resin while still maintaining unaltered the MUF adhesive exterior performance. Thus, from the pure MF resins of about 40 years ago the industry passed to M:U weight ratio of 70:30 and finally of 50:50 and 40:60 for the top performance range of MUF resins used to-day. Resins of lower melamine content also exist, where for example the M:U weight ratio is of 30:70 or even lower, however, their exterior grade performance is definitely and noticeably worse. They are used also but not for the same target applications (Zanetti M. et al., 2003).

From another perspective, there are considerable crude proteins in PKM (Boateng et al. 2008), it has showed the great potential for PKM to work as filler for MUF resin manufacturing. Although wheat flour is used as material to increase the quantity and viscosity of wood adhesive (Kim H. J. et al. 2005), however, this does not increase the bonding strength significantly. Apart from the crude protein content in PKM, PKM also contains 21-23% of crude fibers which is rich in cellulose, hemicelluloses, and lignin. All these crude fibers actually have the similar feature with the wood surface. So, it will facilitate the bonding interactions between the wood adhesives and wood bonding surface. Moreover, the amino groups (-NH_2) in the crude protein of PKM as filler in may form the hydrogen bonding between the melamine and urea in MUF resin and also fibers (cellulose and hemicelluloses) to enhance the bonding strength of MUF resin. In addition, the presence of small amount of lipid in the PKM may also reduce the intrusion of water to disrupt the hydrogen bonding between MUF resin and also the wood surface.

So, to enhance the performance of PKM used as filler for MUF resin, effects of different particle sizes of PKM as filler will be studied in this research. In general, the filler is used as a material to fill up the voids or gaps between the wood adhesive and wood bonding surface in order to increase the contact surface area to maximize the bond strength performance. Particle sizes range in 70-221 μm will be used as filler for MUF resin manufacturing in this study.

Greater adhesive bond strength can be achieved by a prolongation (to a certain limit) of the pressing time, but definitely that will increase the costs of heat energy supplied, and reduce the production capacity (M. Jost et al. 2009). Hence, manufacturers of wood-based panels are continually trying to find an optimal pressing time, which provides a balance between the required performance of the panel and acceptable production costs (M. Jost et al. 2009). Also, the optimum temperature that wood adhesives can achieve maximum and optimum shear strength performance also an important role in determining the production cost and quality of resin.

In practice, trial-and-error methods are frequently used to determine the appropriate press schedule for any particular conditions (Wang and Winistorfer 2003). Mechanical testing of the adhesive bond line is the method used to determine the quality of the adhesive cure and the effectiveness of the wood-adhesive interaction (Steiner and Warren 1981). Thermomechanical analysis (TMA), dynamic mechanical analysis (DMA), torsional braid analysis (TBA), integrated pressing and testing system (IPATES), and automated bonding evaluation system (ABES) are the methods that have been used in examining the mechanical properties of the adhesives during curing. Among these, ABES and IPATES are more preferable in the practical point of view since these techniques provide data on the shear strength of the adhesive bond (ABES) or internal bond (IPATES), whereas TMA, DMA, and TBA measure the changes of the different moduli (M. Jost et al. 2009). ABES is very practical for determination of the shear strength of the adhesive bond and for testing the strength development of this bond during curing (Humphrey 1990). It is because this technique provides valuable data on the shear strength of the adhesive bond as a function of the pressing parameters (e.g., time and temperature) and other conditions (e.g., the cooling effect) (Humphrey 2006).

ABES and similar adapted methods have been found to be useful for determining the development of bond strength for various adhesive types under different pressing conditions (Kreber et al. 1993; Prasad et al. 1994; Chowdhury and Humphrey 1999; Kim and Humphrey 2000; Heinemann et al. 2002a, b; Lecourt et al. 2003; Dunky 2004). However, ABES is a destructive test providing only one data point per test, and thus it has yield the difficulties and numerous works in order to gain optimum and maximum shear strength performance during hot –pressing. So, it is more convenient if there correlation equations can be established to predict the optimum and maximum shear strength operating condition.

Furthermore, none of the previously mentioned mechanical methods can be employed for continuous

testing during the hot-pressing of wood-based composites (M. Jost et al. 2009). Thus, several attempts have been made in order to find prediction equations to predict mechanical testing results that will ease in finding optimum and maximum shear strength performance of MUF resin under different temperature and pressing time. So, Response surface methodology D-optimal has been adopted to aid in finding the correlation equations between temperature, pressing time shear strength performance. In particular, the scope for this study only limited to cover certain range of temperature and pressing time during hot-pressing for MUF resin.

METHODOLOGY

The steps of testing the performance of PKM as filler in MUF resin manufacturing consists of 4 stages of preparation which are: (i) Palm Kernel Meal (PKM) preparation; (ii) Melamine-Urea-Formaldehyde (MUF) resin preparation; (iii) Wood adhesive mixtures preparation; (iv) Wetting, cold press & hot press and (v) Shear strength test . Palm Kernel Meal preparation involves oil removal and also separation of different particle sizes of PKM. Next, MUF resin is prepared according to the formulation of Bono A. et al. 2003 whereas wood adhesive is mixed together with different particle sizes of PKM as filler. Then, the MUF resin with PKM as filler will be applied and spread onto the plywood surface. The plywood will then undergo cold press and hot press, and lastly cut into standard size for performance test according to the Japanese Agriculture Standard.

Palm Kernel Meal Preparation

The palm kernel was obtained from Borneo Samudera Lumadan Palm Oil Mill. The palm kernel was separated from palm kernel shell (PKS), cleaned, washed and air-dried under sun for about 1 hour upon the palm kernel was obtained. The palm kernel was then stored in refrigerator to avoid the fungus from attacking the protein content inside the palm kernel. Next, palm kernel was blended and undergone oil extraction by using soxhlet solid-liquid techniques for about 3 cycles which one cycle was around 4 hours. Iso-propanol was selected as an extraction solvent in the oil extraction process from palm kernel. This extraction process was repeated for three times to make sure the trace oil was completely removed. The oil free palm kernel (palm kernel meal) fiber was then dried in oven about 24 hours at 50^{0}C for removing the trace solvent (Arifin B. et al. 2009).

In order to make particle size classification in more accurate way, PKM with 125-250μm will be sieved through sieve with 221μm and then followed by 150μm pore size, and PKM with 63-125μm will be sieved via sieve with 70μm pore size. The particles sizes of PKM are classified as shown in Table (a).

Table (a) Classification of particle size of PKM

Particle sizes of PKM	Classification
150-221μm	PKM 221μm
70-150μm	PKM 150μm
<70μm	PKM 70μm

Melamine-Urea-Formaldehyde (MUF) Resin Preparation

MUF resin was prepared by three stages. In the first stage, formaldehyde was poured into the flask followed by urea and melamine raw materials. Then the mixture was blended homogenously using a stirrer that connected to a motor as shown in Figure 3.5 and the speed was set to 5. The mixture was a white-colored solution. Temperature of water bath was set to 80^{0}C and the solution pH and temperature were recorded simultaneously from time to time until the end point of MUF preparation (Bono A. et al. 2003). The pH was adjusted between 8.8-9.0 (Pizzi, 1994) in order to prevent the solution from polymerizing too quickly by adding a few drops of sodium hydroxide at 48% concentration into the mixture.

The temperature will gradually increase until it reached 80^{0}C within 50-60 min. At this moment, the pH naturally dropped to below 7.5. The mixture turned clear at about 58^{0}C-60^{0}C. Polymerization process proceeded to a second stage where optimum polymerization of MF resin occurred (Pizzi, 1994) as the temperature reached 80^{0}C and the pH dropped to below 7.5. The end point can be tested by dropping the solution mixture into a beaker of water at 50^{0}C for every 5-10 min. If there is no whitish steak when the droplet is diluted in water means that the end point has not reached yet (Pizzi, 1994). The end point can be detected by using a lower water temperature for a lower degree of polymerization (Bono A. et al. 2003). Stage 3 can be started once the end point was reached, and the pH was adjusted to a range between 8.8-9.5. The resin was allowed to cool down to the ambient temperature and additional urea was

poured into the mixture when the temperature dropped to 65^{0}C (Bono A. et al. 2003). The pH was adjusted to a range between 9.5-9.8 at the end. The cooled resin was then transfer into a plastic container for further testing.

Wood Adhesive Mixtures Preparation

Next, the MUF resin was mixed together with urea, ammonium chloride (hardener) and different particle sizes of PKM fillers as well as in a flour mixer for about 30 min. The well mixed mixture was then applied onto the plywood surface for performance testing.

Wetting, Cold Press & Hot Press

The wood adhesives were later spread and wet on the surface of core and veneers. After the process of wetting, the plywood was then undergoes cold press for 20 min and then followed by hot press with pressing time of 0s, 50s, 150s and 250s for different particles size PKM wood adhesive mixtures at 100^{0}C. Later, the hot press processes with the previous pressing time were repeated again with different particles size PKM wood adhesive mixtures at temperature of 125^{0}C and 150^{0}C respectively.

Shear Strength Test

The shear strength of type II plywood produced here was determined by bonding test according to the Japanese Agriculture Standard (JAS) for structural plywood. For every trial plywood panel produced, total of nine plywood test pieces (25mm x 80mm) were tested. The dimension of plywood test pieces is shown in Figure (a). The test pieces were soaked in a hot water bath at 60 °C for 3 hours and followed by soaking it at cold water bath at room temperature. Once the test pieces reached cold state, shear strength was carried out as shown in Figure 3.8. While the plywood test pieces were wet, the test was conducted using Jiing Koou HT-8311C bonding testing machine (Lisa, 2009).

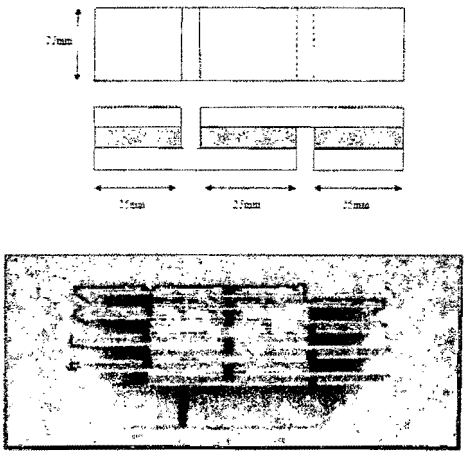

Figure (a) Dimension of Plywood Test Piece for Bonding Test

The shear strength of the test pieces shall be conducted according to the following equation. If the thickness ratio of core veneer to face veneer is a.50 or more, calculated value shall be multiplied by the coefficients in the right column of the following table according to the classes of thickness ratios of the same table and obtain the shear strength. Any plywood panel having the shear strength less than 0.7 MPa is considered fail according to the JAS standard.

$$\text{Shear Strength (MPa or N/mm}^2\text{)} = (Ps \times \text{Earth gravity} \times Co) / (b \times h)$$

where Ps is maximum load (reading from bonding machine),
Earth gravity is equal to 9.81,
Co is co-efficient,
b is width of the piece (mm), and
h is distance between saw cuts (mm).

RESULTS & DISCUSSION

Experimental data analysis with spreadsheet

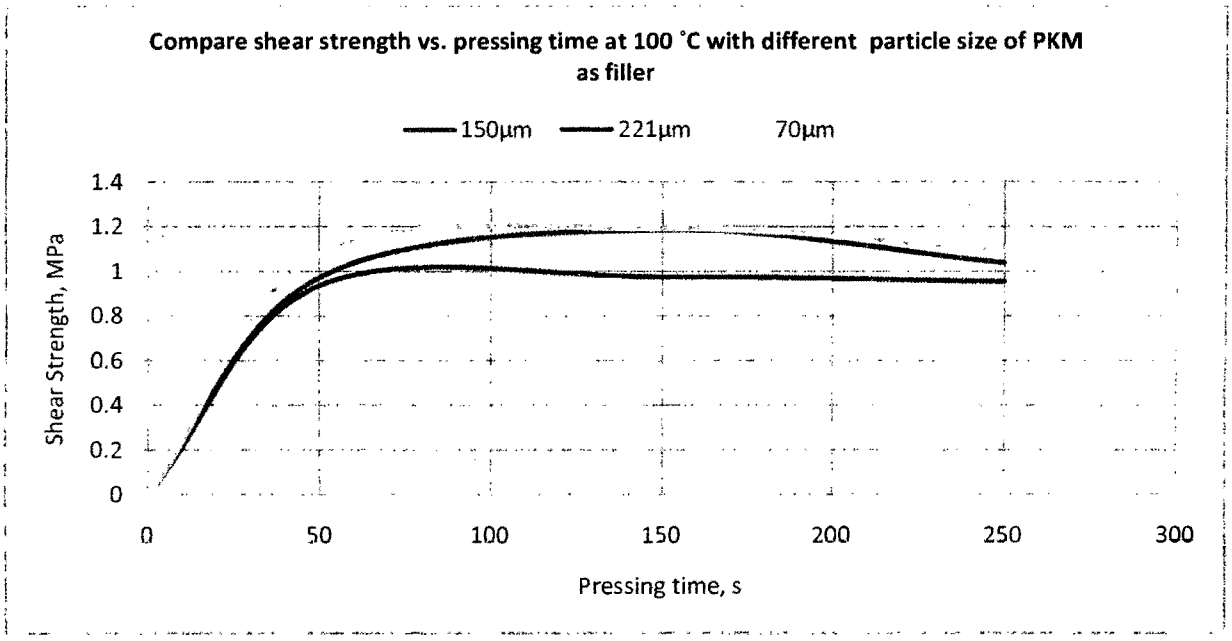

Figure (b) The shear strength growth curve for various particle size of PKM as fillers in MUF resin at 100^0C

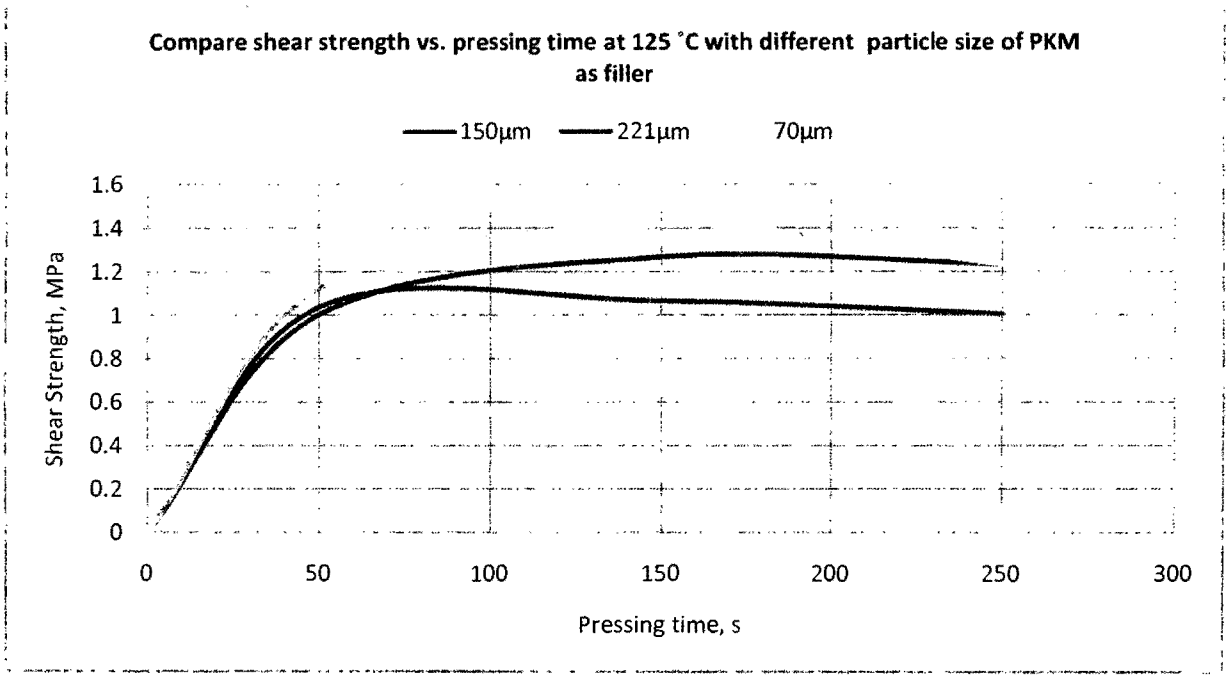

Figure (c) The shear strength growth curve of various particle size of PKM as filler in MUF resin at 125^0C

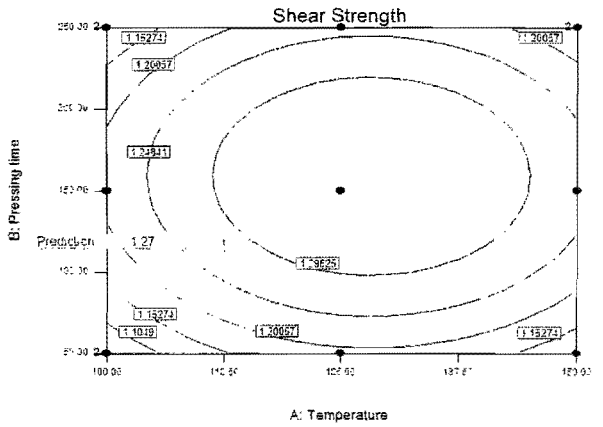

Figure (h) Optimum shear strength PKM 70µm

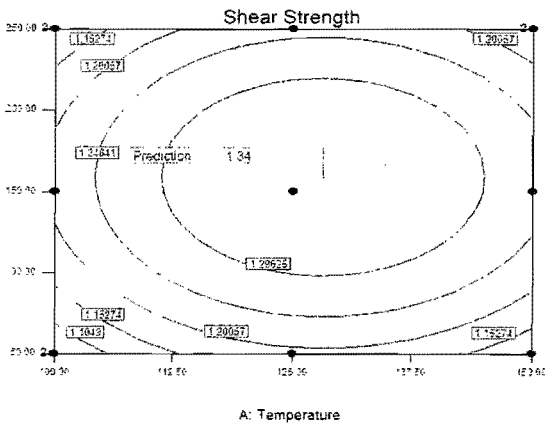

Figure (i) Maximum shear strength PKM 70µm

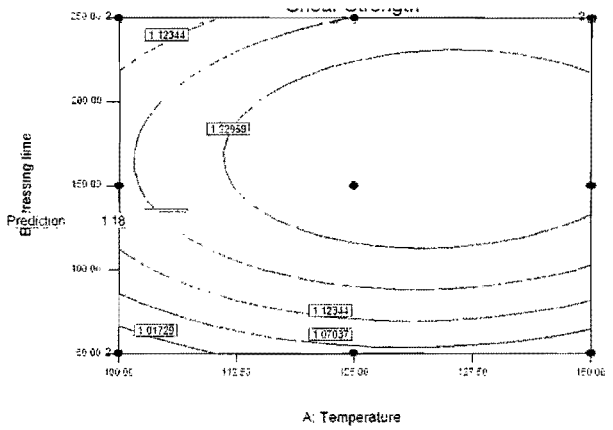

Figure (j) Optimum shear strength PKM 150µm

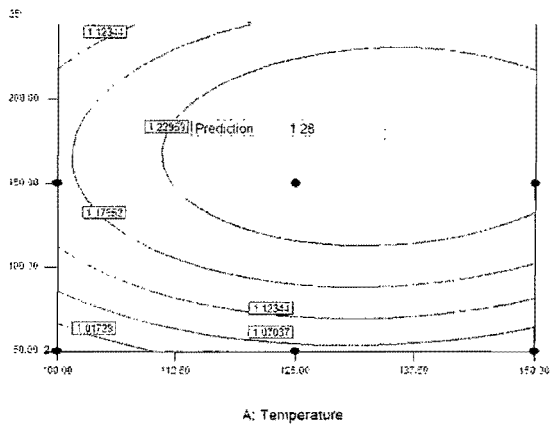

Figure (k) Maximum shear strength PKM 150µm

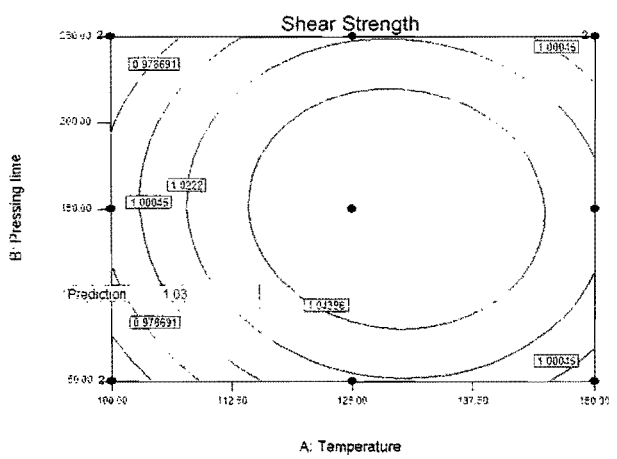

Figure (l) Optimum shear strength PKM 221µm

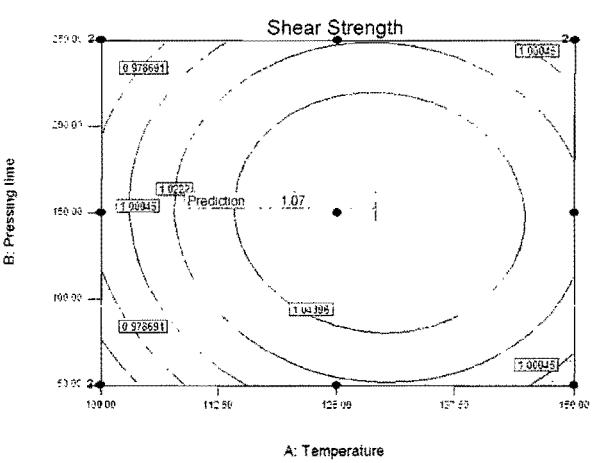

Figure (m) Maximum shear strength PKM 221µm

By comparing Figure (i), Figure (k) and Figure (m), with experimental data from Figure (e)-(g), it is obvious that RSM data and experimental data showed the same trend that larger the particle size of PKM, weaker the shear strength performance. The shear strength of predicted data from RSM showed that descending order: PKM 70μm > PKM150μm > PKM221μm.

Hence, smaller the particle sizes of PKM, higher the chance of particle of PKM to penetrate into the wood surface and fill up the voids on the wood surface and bondlines. Thus, higher the contact surface area of particles of PKM to form bonding with wood molecules. In addition, smaller the particle size of particles of PKM, more intimate contact of adhesive molecules with wood molecules can be built up and thus reduce the number of voids on the wood surface that will disrupt the shear strength performance. From the results, it can be deduced that the best fitness and suitability particle size to the voids on the wood surface is PKM70μm.

Besides, from the mixing process and wetting process observations, particle size of PKM does affect the viscosity and wet ability of MUF resin. Smaller the particle size of PKM, lower the viscosity of MUF resin and thus resulted in better wetting performance. It is because lower the viscosity of MUF resin, lower the surface energy of the liquid of MUF resin. Hence, lower the surface energy, easier the spreading and wetting process and a more even distribution of adhesives molecules on the wood surface.

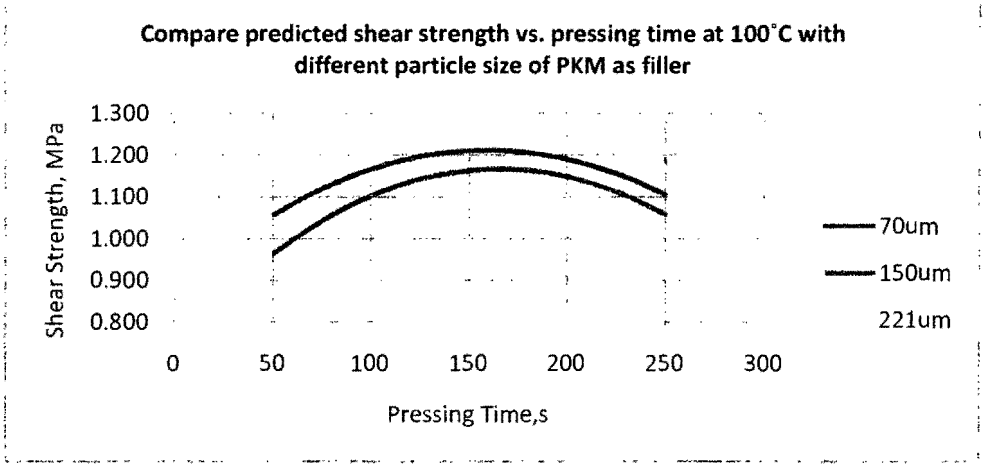

Figure (n) Shear strength performance of various particle size of PKM at 100^0C

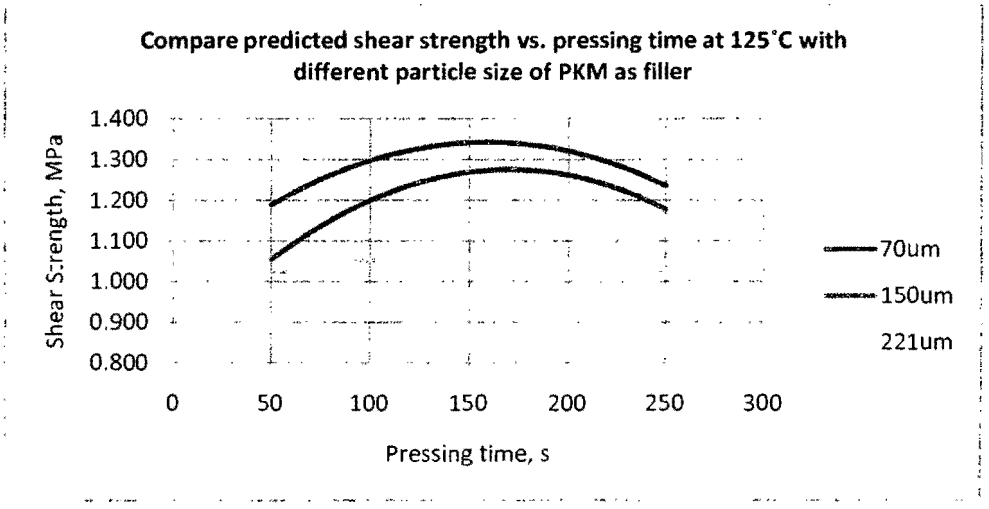

Figure (o) Shear strength performance of various particle size of PKM at 125^{0}C

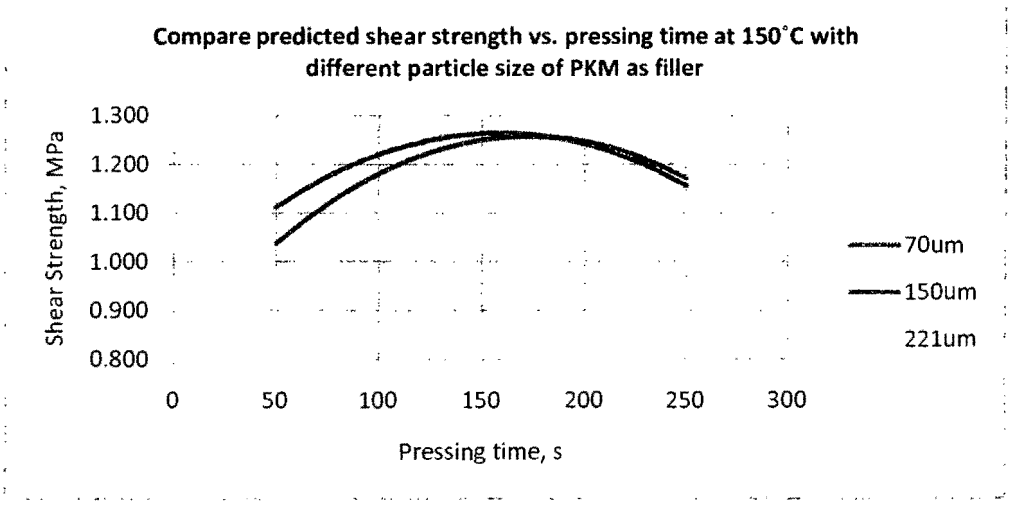

Figure (p) Shear strength performance of various particle size of PKM at 150^{0}C

In most situations, smaller the particles size of the PKM as filler for MUF resin, the predicted shear strength of MUF resin is stronger. These results had been shown in Figure (n), Figure (o) and Figure (p) which were calculated by the equations derived from RSM D-optimal. However, all these equations are limited to specific range of temperature 100-150^{0}C and pressing time 50-250 seconds.

The predicted shear strength performance of MUF resin not solely depends on viscosity and pH of resin, but also the particle size of PKM as filler. PKM 70µm always showed the strongest predicted shear strength performance and followed by PKM 150µm and PKM 221µm. However, when the temperature and pressing time increased to certain level as shown in Figure 4.15, PKM 150µm achieved almost the same predicted shear strength with PKM 70µm. This also similar to the case happened near the end of pressing time in Figure 4.3. This may due to the heat transfer during that particular period was enough for the strong bonding network to be built up between the bondlines and the wood surface and it could overcome the limitation of contact surface area for heat transfer. But, this situation doesn't apply for PKM 221µm, it showed the lowest shear strength for all the times. This may due to the too big the particle size of PKM and unfittable for the gap on wood surface and thus causing the destruction of strong

bonding network to be built up between wood surface and the bondlines.

Due to the considerable crude protein in PKM (Boateng et al. 2008), the hydrogen bonds formation in contributing to the shear strength performance cannot be neglected. It is because the protein consists of significant amount amino groups that can form hydrogen bonds. The effects of hydrogen formation can be seen through Figure 4.14 and Figure 4.15. PKM 70µm showed decreasing in overall shear strength performance when temperature increased from 125^0C to 150^0C. This may due to some protein denaturation happened due to overheating. At particular particles size of PKM as well as 70µm, the heat transfer rate is very high compared others because it has the highest contact surface area. Overheating caused the protein denaturation and subsequently affected the hydrogen bond formation between the bondlines and wood surface. Hence, the shear strength performance of PKM 70µm dropped when temperature is around 150^0C.

However, the effect of increasing temperature towards shear strength performance for PKM 221µm due to destruction of hydrogen bonds formation caused by protein denaturation was not significant. This may due to the low contact surface area of particle molecules with wood surface. In addition, the significant number of voids existed between wood surface and the bondlines which contained air as heat insulator formed a sufficient barrier for delaying the process of overheating to be happened.

CONCLUSION

This study has shown that increasing in temperature and pressing time are not necessary always true in enhancing the shear strength performance of MUF resin with PKM as filler. However, it does depend on the fitness and suitability of particle size of PKM filler to fit into the voids that existed on the wood surface. The results have revealed that the highest maximum and optimum shear strength of MUF resin with fillers is PKM 70µm and then followed by PKM 150µm and PKM 221µm. In general, the shear strength performance of MUF resin dropped after optimum temperature and pressing time. This is due to the protein denaturation in MUF resin with PKM as fillers.

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