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EPOXY ADHESIVES MODIFIED WITH NANO- AND MICRO-PARTICLES FOR *IN-SITU* TIMBER BONDING: EFFECT OF MICROSTRUCTURE ON BOND INTEGRITY

Z. Ahmad[a], *M.P. Ansell*[b] *and D. Smedley*[c]
[a]Faculty of Civil Engineering, Universiti Technologi Mara, Malaysia,
[b]Materials Research Centre, Department of Mechanical Engineering, University of Bath, UK
[c]Rotafix (Northern) Limited, Rotafix House, Abercraf, Swansea, UK,
Email: zakiahah@hotmail.com

ABSTRACT

The bond integrity of thixotropic, shear-thinning epoxy adhesives for *in situ* bonding of FRP connections into timber was investigated. The manufacture of such joints requires adhesives which can produce thick bond-line where the bonding environment does not allow any use of pressure and heat. Four adhesives were evaluated based on a standard adhesive to which nano-silica, nano-rubber and ceramic micro-particles were added to modify rheology and improve adhesive bonding. Viscosity and shear stress of the adhesives were measured as a function of shear rate. The rubber-modified formulation was found to possess the highest viscosity and required higher shear stress to reduce its viscosity. The wettability of nano-modified adhesives on timber substrates was assessed by contact angle measurements and the rubber-modified formulation possessed the highest contact angle which was expected from viscosity measurements. The shear strength of timber to adhesive interfaces was measured using block shear tests and pull-out tests on FRP rods bonded into timber. The addition of nano- and micro-filler additions increased the bond strength of the base adhesive by up to 20%. However the measured contact angle negatively correlated with measured bond strength. Overall, the less ductile micro-particle-filled adhesive delivered the highest shear strengths when bonding timber and FRP rods.

Keywords: micro- and nano-particle-reinforcements, shear strength, wettability, thixotropy.

1. INTRODUCTION

Bonded-in timber connections are effective in jointing timber without the use of mechanical fasteners and bolts (Ansell and Smedley, 2008). Rods or plates made from pultruded fibre reinforced plastic (FRP) or metal are bonded into timber using structural adhesives. The adhesives are back-injected into oversize holes or slots and the rods or plates are pushed into the adhesives forming a void-free interface and the adhesive is allowed to cure. The bond integrity influences the ultimate capacity of the bonded structure as well as serviceability and depends on a number of factors including the type of adhesive and adherend, cure cycle, bondline thickness and the environment. The bond strength depends on how well the adhesive wets the adherends and the lower the viscosity, the more easily the adhesive will penetrate the adherend. Carpenter (1991) provides an overview of strength theories for the design of adhesively bonded lap joints with composite adherends and numerous references review adhesive bonding of timber (Kinloch and Young, 1983, Davis, 1997, Pizzi and Mittal, 1994). However, these references focus on low viscosity adhesives which require pressure and high temperatures for curing. In order to achieve effective room temperature *in-situ* bonding of pultrusions into timber without the application of pressure, the adhesives used must be thixotropic and shear thinning so that they cannot run out of inverted holes.

Research on timber connections based on bonded-in rods has assessed variations in the rod type and size, glue-line thickness, type of timber and geometry of the connection (Joseph, 1999, Davis and Claisse, 2001, Broughton and Hutchinson, 2001, Harvey and Ansell, 2000). Less attention has been paid to the properties of the adhesives used and their effect on joint integrity. Therefore this study investigates the properties of three room temperature curing adhesives containing nano-particles which are thixotropic and shear thinning, allowing injection into overhead holes. A fourth formulation is micro-particle-filled resulting in a higher modulus of elasticity and different rheology. The adhesives are formulated with different additions of nano-particles and micro-particles in order to optimize the strength of bonded-in timber connections. The properties of the uncured adhesives are assessed by performing rheological tests with a rheometer and contact angles are measured on laminated veneer lumber (LVL) substrates to evaluate wettability. A block shear test is used to evaluate the adhesive to LVL bond strength and pull-out tests are performed on pultruded GFRP rods bonded into LVL. The contact angles were also determined. Fracture topography following block shear and pull-out tests is evaluated using scanning electron and optical microscopy.

2. METHODOLOGY

2.1 Materials

Four types of thixotropic and room temperature cured epoxy-based adhesive obtained from Rotafix Ltd were used in this study, referred to here as CB10TSS, Nanopox, Albipox and Timberset. CB10TSS is a mixture of a diglycidylether of bisphenol-A (DGEBA), a reactive monofunctional diluent glycidlyether, silica fume particles, hardener, a mixture of polyetheramines and other trade secret ingredients. CB10TSS is a commercial product and is considered to be the standard thixotropic adhesive. The other three adhesives were formulated by modifying CB10TSS with the addition of either nanosilica (Nanopox), a carboxyl-terminated butadiene acrylonitrile liquid rubber (Albipox) or a mixture of bentonite, quartz and mica micro-particles (Timberset). The thixotropic nature of the adhesives evaluated in this research is controlled by the reactive diluent content together with the nano- and micro-sized additives employed. Timberset is heavily filled with micro-particles resulting in a high stiffness cured adhesive with a Young's modulus of ~11GPa compared with ~3GPa for the other formulations which are considerably more ductile.

2.2 Preparation of specimens and experimental measurements

2.2.1 Viscosity measurements

Rheological tests were carried out by employing a cone and plate method using a Bohlin CS Rheometer at various shear rates at 25°C. The angle and radius of the measuring cone were 4° and 20 mm respectively. The height of the tip of the cone and the plate was set at 0.15 mm.

The adhesives were prepared in accordance with the manufacturer's instructions with due regard given to handling precautions for the adhesives. The two-part adhesive was prepared by mixing the base adhesive and the hardener using a rotary stirrer at constant speed for 5 minutes. The adhesive was left to rest for 2 minutes before placing a small amount of adhesive on the plate sufficient to fill the gap between the upper and lower elements. Shear stress and viscosity were recorded as a function of shear rate.

2.2.2 Contact angle

Contact angles were measured using a Contact Angle Goniometer model 100-00. Small LVL blocks of size 10 mm thick x 30 mm width and 50 mm length were cut from an LVL beam so that the veneer was exposed on the top surface which was cleaned with a dry cloth. The LVL block was placed on the specimen stage of the Goniometer. Droplets of adhesive of 0.5 ml in volume (Figure 1a) were delivered onto the LVL surface. The contact angle was measured with a microscope within five minutes after deposition as shown in Figure 1b. The measuring crossline was adjusted so that it attained tangency with the drop profile at the base of the drop. The contact angles recorded are the mean values for droplets placed on 5 blocks with at least 3 droplets per block (Figure 1c).

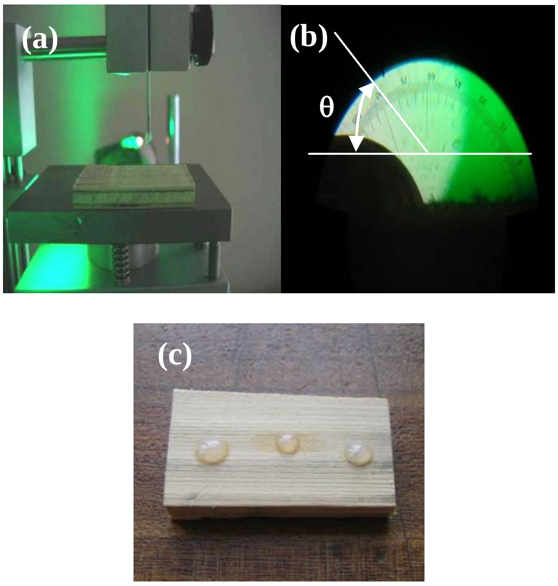


Figure 1: Showing; (a) Sessile droplets of CB10TSS adhesive, (b) measurement of contact angle, θ. and (c) LVL blocks with droplets of adhesives.

2.2.3 Block shear test

Block shear specimens were prepared from 50 mm thick Kerto LVL panels manufactured from Norwegian spruce. Prior to that, three 50 mm x 50mm 50 mm blocks were cut from the LVL panels for moisture content measurements. The remaining LVL panel was sliced into two parts in the plane of the veneer (avoiding glue lines between veneers) to make two beams, one 22 mm thick and the other 25 mm thick. The LVL adherends were laid side by side. Two Perspex shims of 3 mm thickness were placed at the end of the first adherend as thickness guides. An aluminium plate was attached with brown tape to the bottom of the adherend to prevent the adhesive from leaking out. The adhesive was poured and spread slowly onto the adherend. Then the second adherend was placed on top of the adherend that contained the adhesive. The wood composite was clamped together by binding it with brown tape and also by using steel clamps to secure the bonding. The composite was left to cure at approximately 20° C and 65% RH for 20 days and then the LVL adherends were trimmed at the edges to remove the aluminum plate and the shims. The beam specimens were cut into cubes with 50 mm edge dimensions (Figure 2). The LVL and adhesive thickness were chosen so that one adhesive to LVL interface was subjected to shear. Ten replicates were randomly chosen per adhesive formulation. The test specimens were stored in a humidity cabinet until the time of testing. The shear specimens were tested with a shear fixture according to ASTM D 905. A compressive load was applied using an Instron 1185 test machine with a 100 kN load cell at a crosshead rate of 0.5 mm/min. The shear strength was calculated at the maximum load.

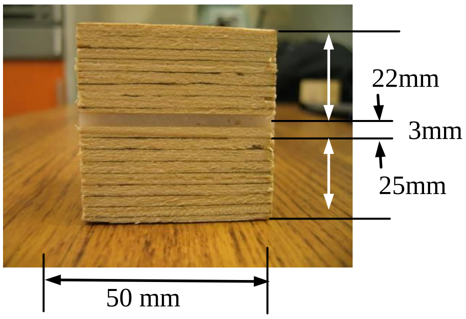


Figure 2: Block shear test specimen.

2.2.4 *Pull-out of bonded-in rods*

LVL blocks were prepared with edge dimensions of 63mm and a central hole was drilled on one face to make 14 mm diameter holes parallel to the grain. The inner surface of the hole was lightly sanded in order to improve bonding by the removal of loose wood fibre. The glass fibre reinforced plastic rod of 8 mm diameter was also lightly surface abraded and cleaned with ethanol before inserting into the hole back-filled with adhesive resulting in a 3 mm annular bond line thickness (See Figure 3a). A rubber O-ring was placed at the top and bottom of the hole in order to centre the rod. The bonded-in timber specimens were left to cure in a controlled humidity room (20° C and 95% RH) for 20 days.

After conditioning the specimens were tested in tension using an Instron 1185 test machine with a 100 kN load cell, using a steel and aluminium cage developed in previous work (Madhoushi and Ansell, 2004) (see Figure 3b). The timber blocks were positioned within the cage, with the rod protruding through a hole machined in the base plate of the jig. The base of the cage acted as a reaction plate against the timber block as the rod was pulled through the hole at a constant cross-head displacement rate of 2 mm/min.

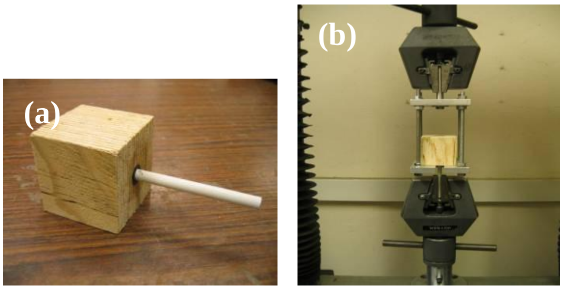


Figure 3: (a) Single-ended pull-out test specimen and (b) Experimental set-up for pull out test.

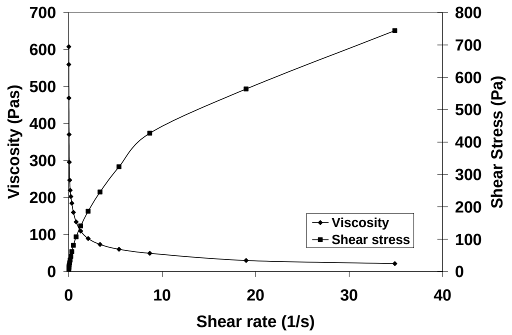
2.2.5 *Scanning electron and optical microscopy*

After gold coating the fracture surfaces of the block shear specimens and bonded-in rods were inspected in a JEOL JSM6310 scanning electron microscope (SEM) equipped with an image analysis system. Optical microscopy was employed to examine failure in pull-out specimens.

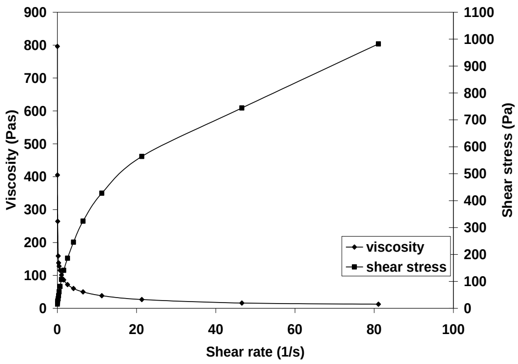
3.0 RESULTS AND DISCUSSION

3.1 Rheological properties

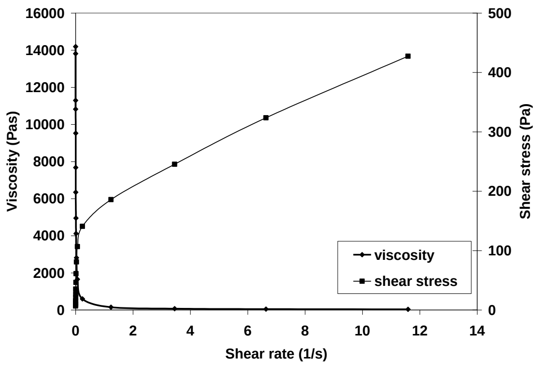
In order to examine the effect of micro- and nano-fillers on the viscosity of the adhesive formulations, rheological measurements were carried out at 25° C in a Bohlin viscometer. The viscosity values for the CB10TSS, Nanopox and Albipox are presented in Figure 4 for different rates of shear and the values recorded were confirmed as valid by the Bohlin software.



(a)



(b)



(c)

Figure 4: Viscosity and shear stress plotted versus shear rate for (a) CB10TSS, (b) Nanopox and (c) Albipox.

From these graphs it is evident that the viscosity of each adhesives decreases with increase in shear rate, i.e. each adhesive behaves like a non-Newtonian fluid. In order to confirm that the adhesive behaves thixotropically the adhesives need to be subjected to increasing and decreasing shear to generate a hysteresis loop.

However this was not done due to the limitations of the equipment. However by observation and during handling, the CB10TSS was found to be thixotropic in practical use. The viscosity of Timberset was not determined as it has a very high viscosity and crosslinks rapidly so damage to the equipment would have occurred. The time for the shearing to take place at a constant rate of increase in shear rate varied from one adhesive to another. CB10TSS is much easier to shear compared to Nanopox and Albipox as it required less shear stress to reduce its viscosity. The Albipox matrix contains a rubbery phase and showed the least pronounced pseudoplasticity (i.e. the viscosity decreasing with increasing shear rate) and it had the highest viscosity at low shear rates, indicating physical interaction between the epoxy and the rubbery phases. It is possible that cavitation or debonding of rubber particles took place which increased the resistance of the adhesive formulation to flow under shear.

3.2 Wetting properties of the adhesives

The contact angle between a droplet of the adhesive and the timber surface was measured using a goniometer. The contact angle measurements for CB10TSS, Nanopox, Albipox and Timberset are tabulated in Table 1. The contact angle measurement for Timberset could not be obtained because the adhesive is highly viscous and a sessile drop cannot be formed. Table 1 shows that Albipox has the highest contact angle values followed by Nanopox and CB10TSS and these results correlate well with the viscosity measurement in Section 3.1.

Table 1: Contact angles measurement for the adhesives.

Adhesive	Contact angle (°)	Std. Dev.
CB10TSS	58.6	5.8
Nanopox	46.6	8.9
Albipox	72.3	12.3

3.3 Block shear test

The average shear strengths obtained from block shear tests at room temperature are listed in Table 2. It is clear that the mean shear strength for Timberset is the highest followed by Albipox, Nanopox and CB10TSS.

Table 2: Shear strength of adhesive-timber joints.

Adhesive	No. of sample	Shear strength		Failure mode
		Mean (MPa)	Std. Dev	
CB10TSS	10	5.7	0.3	Timber
Nanopox	11	6.1	0.9	Timber
Albipox	10	6.4	0.4	Timber
Timberset	10	6.9	0.6	Timber
*LVL	10	4.9	0.2	Bond-line interface

Note: * shear strength of LVL specimens alone.

The mean shear strength of Timberset which contains ceramic micro-particles is 21% higher than the standard adhesive, CB10TSS. The mean shear strength of Albipox is 12% higher than CB10TSS. The ranking order of the shear strength values for adhesive joints contradict the ranking order of the contact angle as mentioned in Section 3.2. It is expected that low contact angles correspond to better wetting and hence a higher shear strength. Despite having a higher contact angle, block shear samples bonded with Albipox still possess a higher mean shear strength value compared to CB10TSS. Timberset has a higher viscosity than Albipox at room temperature and yet it has higher mean shear strength. Therefore, from these observations, it appears that high viscosity adhesives with high contact angles possess higher bond strengths. The wetting of the LVL surface is not so much of an issue as failure occurs in the LVL adjacent to the bond-line as described in Section 3.5. All the specimens failed in the timber close to the interface between the adhesive and the timber, indicating that there is a good adhesion and wetting between the adhesive and timber.

There is no significant difference in shear strength values for CB10TSS and Nanopox so no further investigations were undertaken on Nanopox adhesive. An improvement in adhesive bond strength has been reported by many authors using CTBN liquid rubber as a modifier (Bascom and Cottington, 1976, Huang *et al*, 1993). Achary *et al* (1991) reported a three-fold increase in lap shear strength using carboxyl terminated poly (propylene glycol) adipate as liquid rubber. Ratna and Banthia (2000) reported a two-fold increase in lap shear strength using carboxyl-terminated poly (2-ethylhexyl acrylate) (CTPEHA) as the liquid rubber. According to these authors, the enhancement of bulk mechanical properties, hence the adhesive joint strength was attributed to the higher toughness produced by the dispersed rubber particles. These observations concur with shear strength results for Albipox.

The shear tests results for bonded interfaces were also compared with block shear results for LVL specimens alone (Table 2). LVL is made from laminated 3 mm thick spruce veneers with very thin phenolic resorcinol formaldehyde bond-lines. The shear strength of the LVL is significantly less than the LVL specimens with thick bond-lines. The shear stress is applied in the plane of the wood to adhesive interface in both specimens. For LVL alone, the failure was an adhesive failure and for LVL with thick bond-line nano-particle filled adhesive, the failure was cohesive timber failure. Hence it is likely that the thixotropic adhesives have better adhesion with timber surface compared to PRF adhesive, raising the shear strength.

3.4 Pull-out test

The pull-out strength of bonded-in rods was used to investigate the effect of nano- and micro-filler additions on the strength of bonded-in timber connections. This test most closely simulates a bonded-in connection under load. The failed pull-out test specimens show three different failure modes (Figure 5).

The average shear stresses at the adhesive to timber and rod to adhesive interfaces were calculated by dividing the failure load by the bond area using Equations [1] and [2]:

Adhesive-timber interface

$$\tau_{ta} = \frac{P_{\max}}{\pi \phi_{hole} L} \qquad [1]$$

and rod-adhesive interface

$$\tau_{ra} = \frac{P_{\max}}{\pi \phi_{rod} L} \qquad [2]$$

where ϕ_{hole} and ϕ_{rod} are the diameters of the drilled hole and rod respectively and L is the rod embedment length which was 65 mm in all cases. Due to the different diameters, the bond area at the adhesive/timber interfaces is bigger than the bond area at the adhesive/rod interface which results in different strength values.

Figure 5: Different modes of failure; (a) timber failure (t), (b) rod/adhesive failure (ra) and (c) timber/adhesive failure (ta).

Ten specimens were tested for each adhesive type and Table 3 summarises pull-out test results. An analysis of variance was performed for the shear strength at the rod-adhesive interface (denoted as shear strength$_{ra}$) and shear strength at the timber-adhesive interface (denoted as shear strength$_{ta}$).

The ANOVA reveals that there is significant difference in the shear strength$_{ra}$ (p-value=0.000) and shear strength$_{ta}$ (p-value=0.000). From DUNCAN multiple comparisons it was found that there is no significant difference in the shear strength$_{ra}$ and shear strength$_{ta}$ for CB10TSS and Albipox and both of them failed 100% at the rod-adhesive interface. There is significant difference in the shear strength$_{ra}$ values and shear strength$_{ta}$ values between Timberset and CB10TSS and between Timberset and Albipox. The pull-out shear strengths of bonded-in rods for Timberset were higher than for Albipox and CB10TSS.

Table 3: Shear strength of pull-out specimens.

Adhesives	Pull-out force		Shear stress at rod to adhesive interface		Shear stress at timber to adhesive interface		Failure Mode (ra, ta, t, rat)*
	P_{max} (kN)	COV (%)	τ (MPa)	COV (%)	τ (MPa)	COV (%)	
CB10TSS	11.1	1.6	7.0	1.1	4.0	0.8	100% ra
Albipox	11.5	1.8	7.3	1.2	4.2	0.7	100% ra
Timberset	17.4	1.7	11.0	0.7	6.3	0.4	~40% ra, ~40% t, ~20% rat

* ra - rod/adhesive failure, ta - timber/adhesive failure,
t – timber failure, rat – rod/adhesive/timber failure

The results from the experimental tests show that the pull out shear strength at the rod/adhesive interface for all adhesives exceeds the shear strength of the LVL (~5MPa). Once again the bond strength of the low viscosity adhesive is less than the bond strength of the high viscosity adhesive.The difference in the shear strength values for the CB10TSS, Albipox and Timberset adhesives depends on chemical and mechanical bonding and is reflected in the failure modes. To further investigate the joint interfaces, optical microscopy was used to examine the rod/adhesive and adhesive/timber interfaces following pull-out tests and the micrographs are examined in Section 3.6.

3.5 Fracture surfaces following shear testing

Pizzi (1983) states that "for two adherends to be held together with maximum strength, the adhesive must flow onto and wet the adherends before the adhesive reaches a rigid state and produce an intimate contact. Molecules of the adhesive must diffuse over and into each surface to make contact with the molecular structure of timber so that the intermolecular forces of attraction between adhesive and timber can become effective.

Failure surfaces following block shear testing of all the adhesives were similar with failure predominantly in the timber and only a few blocks with a small percentage of the surface failed at the adherend-adhesive interface which can be regarded as cohesive failure in timber. Examples of such surfaces are shown in Figure 6a & b respectively.

The type of failure is an important indicator of bond strength complementing the measured shear strength of the bond. In order to examine the process of failure within a block shear specimen, the shear failure surface of a small block shear specimen was gold coated and observed by SEM.

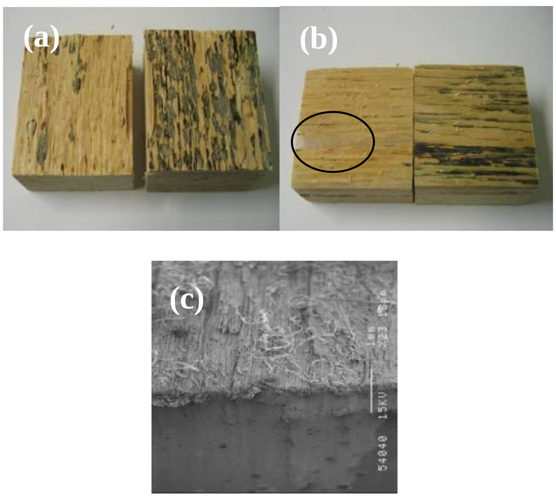


Figure 6: Failure mode of block shear sample: (a) for CB10TSS: cohesive timber failure, (b)for Nanopox: crack propagation in the timber with small part failed at the adhesive-timber interface (indicated by the marked circle) and (c) for CB10TSS**:** SEM image obtained from the shear failure surface showing the high proportion of timber failure.

Figure 6c shows that the fracture propagates in the timber as wood fibre is found on the adhesive surface of the failed joints (also see Figure 6b). The higher the proportion of timber failure and the deeper the fracture surface into the grain of the wood, the stronger the bond is (Vick, 1999).

The amount of adhesive penetration into a wood substrate has a direct correlation to the bond quality (Chandler *et al*, 2005). To observe the bonded interface between the adhesive and timber adherend, the unfailed block shear specimen for CB10TSS, Albipox and Timberset was sectioned to reveal the adhesive to adherend interface as shown in Figures 7a, 7b and 7c respectively. The adhesive can clearly be distinguished from the timber (Figures 7a, 7b, and 7c).

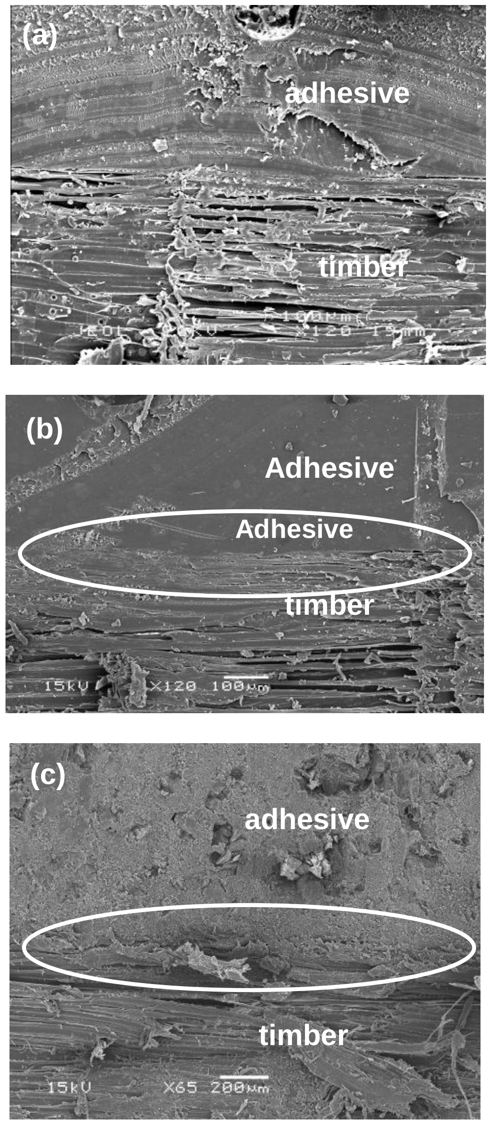


Figure 7: SEM micrographs of the sectioned surface of block shear specimens of (a)CB10TSS, x 120, (b) Albipox, x 120, the marked circle shows smooth bond-

line and (c) Timberset, x 200, the marked circle shows the rough bond-line.

The aim was to evaluate adhesive penetration into the timber. Adhesive penetration, defined by Sernek *et al* (Sernek *et al*, 1999), is the spatial distance into the timber from the interface of the adjoining substrate. As defined by Brady and Kamke (1988), the volume containing the wood cells and adhesive is the interphase region of the adhesive bond. The depth of penetration of the adhesive determines the size of the interphase region. In this study the depth of penetration of the adhesive into the timber was not measured quantitatively as described by Sernek *et al* (1999).

However it can be seen that the bond integrity in CB10TSS (Figures 7a) and Albipox (Figure 7b) appears to be good where an intimate bond line can be seen at the timber to adhesive interface but with the absence of a mechanical interlocking mechanism. The bond-line for Timberset (Figure 7c) appears to rough with the appearance of zigzag line. Although the CB10TSS and Albipox bonded joint appears to be well bonded, its mean block shear strength is lower than the values for Timberset. The rough bond-line (the zigzag line) may be the indication of mechanical interlocking which induce the high value of shear strength in Timberset. Chandler *et al* (2005) in their examination of adhesive penetration in timber using fluorescence microscopy found that good adhesive penetration into lumens does not always relate to bond strength or the percentage wood failure.

The bond strength and the fracture strength of adhesive joints depend on a number of factors and their combinations, e.g. adhesive type, cure cycle etc. Adhesive bonding takes place when an adhesive undergoes a conversion from liquid to solid. The liquid properties are needed for the adhesive to fully wet the bonding substance, and the solid properties are needed for the strength required for the union of the final product. The mobility of an adhesive depends heavily on its own physical and chemical properties and those of the wood surface upon which it is being applied. CB10TSS, Albipox and Timberset adhesives have different curing rates and viscosities and these may affect the bond performance in a manner which cannot be explained here.

Adhesion results from intimate intermolecular contact between two materials and involves surface forces that develop between the atoms in the two surfaces. The wetting involves a reduction of interfacial energy and Huntsberger (1981) showed that the free energy always decreases upon liquid/solid contact. In this investigation, the prepared surfaces of timber adherence were from same timber, dry and parallel grain (see Figure 1c). The adhesives used having same based-adhesives but with different addition of nano-particles. Therefore differences in the bond strength may be caused by different interfacial energy associated with the addition of nano-particles on the surface wetting.

3.6 Fracture following pull-out

SEM micrographs of fractured pull-out test specimens are presented in Figures 8 to 10. From Table 3, it can be seen that Timberset failed in three modes; at the rod-adhesive interface (ra), in the timber (t) and in a combined rod-adhesive-timber mode (rat) while Albipox and CB10TSS failed mainly at the rod to adhesive interface (ra) which reflects the better interfacial bond between rod and Timberset as seen in Figures 10.

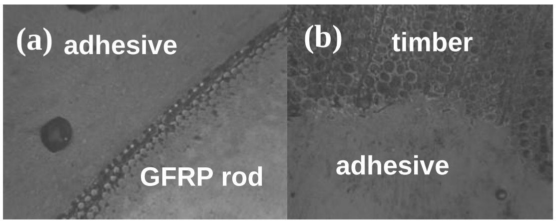


Figure 8: Optical micrographs of failed pull-out specimens for CB10TSS: (a)Joint between the GFRP rod and adhesive and (b) Joint between adhesive and timber.

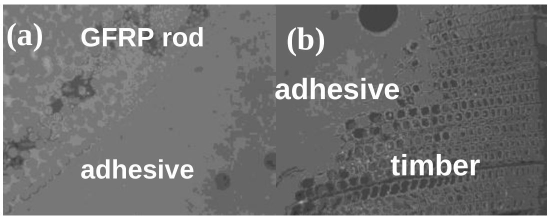


Figure 9: Optical micrographs of failed pull-out specimens for Albipox: (a)Joint between the GFRP rod and adhesive and (b) Joint between adhesive and timber.

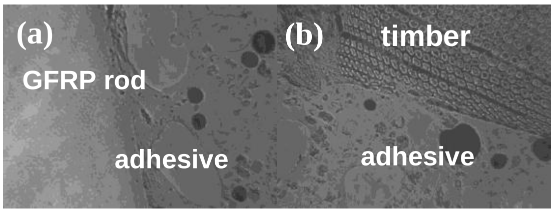


Figure 10: Optical micrographs of failed pull-out specimens for Timberset: (a)Joint between the GFRP rod and adhesive and (b) Joint between adhesive and timber.

Timberset, a highly viscous adhesive, bonds well with the timber surface and the surface of the GFRP rod supporting the observation made in Section.3.2. The low shear strength for CB10TSS and Albipox is reflected by the poor adhesion between the rod and adhesive as seen in Figure 8a. Even though there is no significant different in the shear strength for CB10TSS and Albipox, the adhesion between the rod and adhesive for Albipox appeared to be better as compared in Figures 8a and 9a. In Fig. 9b for Albipox some of the adhesive penetrates the timber cell lumens. This shows that there is a good bond between Albipox and the wood but the low shear strength is due to weak bonding between Albipox and the rod (see Table 3). For bonded-in timber connections, it is preferable for fracture to occur in the timber away from the glue-line interface since the area of shear failure is higher. Further investigation is required to improve bonding between both CB10TSS and Albipox and the rod surface.

In a previous study by the authors (Ahmad *et al*, 2006), the CB10TSS adhesive was shown to be more ductile than Albipox and Timberset was seen to be a brittle adhesive. Differences in rheology have a significant effect on the bonded joint. Feligioni *et al* (2003) worked on the influence of ductile and brittle epoxy adhesives on the shear strength of bonded-in steel rods. Unlike the results reported here the authors found that the shear strength of interfaces using ductile adhesives was higher than for brittle adhesives. However, the shear strength of the ductile adhesive reduces as further cross-linking occurs while the brittle adhesive is more stable. The same observation was found in previous study by the authors [Ahmad *et al*, 2006] where the Timberset cross-links far more rapidly than CB10TSS and Albipox. The difference between the results of Table 3 and Feligioni *et al* (2003) mainly to different materials used where these authors used steel threaded rod which provides mechanical anchorage as compared with the GFRP rods used in this study were without threaded. Further research is needed to improve the adhesion at GFRP rod/adhesive interface.

4.0 CONCLUSIONS

In the measurement of viscosity during cure, CB10TSS is much easier to shear compared to Nanopox and Albipox. Therefore the addition of silica nano-fillers to Nanopox and rubber nano-fillers to Albipox increases resistance to flow in shear. Lower viscosity adhesives are expected to wet surfaces better, increasing the bond strength. In contact angle measurements, Albipox has the highest contact angle values followed by CB10TSS and Nanopox, which correlates well with viscosity measurements. However for Albipox, Nanopox and CB10TSS higher contact angles resulted in higher shear strengths which contradict the contact angle measurements. No contact angle measurements were obtained for Timberset due to its high viscosity but this micro-particle filled formulation achieved higher block shear strengths than the other nano-reinforced adhesives. This trend is supported by the results obtained in pull-out tests where Timberset has the highest pull-out strength followed by Albipox and CB10TSS. In pull-out tests, all CB10TSS and Albipox samples failed at the rod/adhesive interface but Timberset samples failed in a combination of modes in the timber, at the rod/adhesive interface and at the rod/adhesive/timber interface. This indicates that the high viscosity micro-particle-filled adhesive bonds well with GFRP and timber. Hence the lower modulus, more ductile Albipox, Nanopox and CB10TSS adhesives do not perform as well as Timberset for bonding timber and pultrusions despite Timberset's more brittle micro-particle-filled structure. However Timberset has less shear-thinning characteristics as compared to CB10TSS and Albipox in its uncured state so it is less suited to the more critical demands of *in situ* bonding of connections in timber structures.

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