



Material properties

Investigation of bulk and *in situ* mechanical properties of coupling agents treated wood plastic compositesYonghui Zhou ^{a, b}, Mizi Fan ^{a, b, *}, Lanying Lin ^b^a College of Materials Engineering, Fujian Agriculture and Forestry University, PR China^b Department of Civil Engineering, College of Engineering, Design and Physical Sciences, Brunel University, London, UB8 3PH, United Kingdom

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ABSTRACT

This paper presents the interfacial optimisation and characterisation of WPC by the use of maleated and silane coupling agents (MAPE, Si69 and VTMS), and its effect on the bulk and *in situ* mechanical properties. The results showed the treated WPC possessed better interface by showing improved compatibility between the constituents, wettability of wood flour, and resin penetration in the SEM images. The enhanced interface led to the increase in the tensile strength and stiffness of the treated WPC, which was confirmed by their superior load bearing capacity, namely the higher storage moduli measured by DMA. The observed shift of the relaxation peak of the treated WPC indicated the constraints on the segmental mobility of the polymeric molecules resulted from the treatments. Nanoindentation investigation revealed that the *in situ* mechanical properties were subject to a number of phenomena including fibre weakening or softening impact, crystalline structure transformation and cell wall deformation, concluding that the bulk mechanical properties of WPC might not be governed by the local property of materials within the interface.

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1. Introduction

Wood plastic composites (WPC) has recently experienced considerable expansion, mainly because of the advantageous features that wood material possesses, namely ubiquitous availability at low cost and in a variety of forms, renewability and biodegradability, low density, nontoxicity, flexibility during processing, and acceptable specific strength properties [1–7]. However, the formulation of WPC was perturbed by the inherently polar and hydrophilic nature of wood flour or fibre, which makes it least compatible with hydrophobic polymeric matrices. The poor combination of wood and polymer is not able to generate the designed performance [4,8]. To avoid these drawbacks, various specific structural modifications approaches have been studied, such as corona treatment, plasma treatment, UV and gamma radiation treatments, surface compatibilisation and copolymerisation [8,9].

The performance and behaviour of WPC was not only relying on the reinforcing wood and the polymer matrix, but also critically depending on the effectiveness of load/stress transfer across the

interface [10,11]. The interface of WPC formed when the wood flour is embedded in the polymer matrix during fabrication of the composite is a heterogeneous transition zone with distinct chemical compositions, morphological features and mechanical properties from those of the reinforcing phase and the bulk polymer [11–13]. An appropriately engineered interface could considerably improve the strength and toughness of the composite as well as the environmental stability. Therefore, on the other hand, the determination of interfacial properties and characteristics would be of utmost importance in evaluating the overall property of the composite and enabling its optimal design [14]. Numerous techniques, such as single fibre fragmentation test, single fibre pull-out test and microbond test, have been developed for characterising the fibre/matrix interfacial strength. However, very few studies have carried out the determination of the size and relative mechanical properties of the interface due to the lack of unequivocally established experimental techniques.

Nanoindentation technique has been proven to be an effective method in determining material surface properties at nanoscale, which is achieved by monitoring a probe penetrating into the specimen surface and synchronously recording the penetration load and depth [15,16]. It has recently found its application feasible to wood, natural fibres and plastics [12,15,17–24], but it has been

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rarely employed in measuring the interface property and performance of WPC materials. A study on the effect of water absorption on the nanomechanical properties of woven fabric flax fibre-reinforced bioresin-based epoxy biocomposites observed approximately 35% and 12% deductions of nanohardness and reduced modulus respectively, after exposure to water, indicating that the strength and elastic modulus were fibre-sensitive properties in the composites and the interface suffered from the water absorption [25]. Nanoindentation was also conducted to measure the hardness and elastic modulus of in the interface region of cellulose fibre-reinforced PP composite. There was a gradient of hardness and modulus across the interface region, and the distinct properties were revealed by 1–4 indents depending on the nanoindentation depth and spacing [12].

The incorporation of silane and maleated coupling agents exerted reinforcing impact on the global property of WPC materials, but its influence on the mechanical properties in the interface region has not yet been reported in the literatures. In this work, WPC materials were fabricated by the use of recycled wood flour and PE aiming at reducing the consumption of virgin raw materials and the environmental impact. The focus of this work was to optimise the interface of WPC by incorporating three different coupling agents, i.e. maleic anhydride grafted polyethylene (MAPE), bis(triethoxysilylpropyl)tetrasulfide (Si69) and vinyltrimethoxysilane (VTMS); hence to reveal the interface structure and bonding scenarios of the formulated WPC. More importantly, the influence of the coupling agent treatments on the *in situ* mechanical properties of coupling agent treated WPC was first determined by carrying out nanoindentation analysis, which led to thorough understanding of interfacial characteristics and the correlation between *in situ* and bulk mechanical properties.

2. Materials and methods

2.1. Materials

Recycled wood flour used in this work was supplied by Rettenmeier Holding AG (Germany), with a bulk density of 0.285 kg/m³; recycled polyethylene (PE) pellet with a bulk density of 0.96 kg/m³ and MFI of 0.6 g/10 min at 190 °C was obtained from JFC Plastics Ltd (UK); lubricants 12-HSA (12-Hydroxyoctadecanoic acid) and Struktol TPW 709 were purchased from Safic Alcan UK Ltd (Warrington, UK); coupling agents, MAPE (500 cP viscosity at 140 °C, 0.5 wt% of maleic anhydride), Si69 (>95% purity, 250 °C boiling point) and VTMS (>98% purity, 123 °C boiling point), were purchased from Sigma-Aldrich (Dorset, UK), and their chemical formulae were presented in Fig. 1. All the raw materials and additives were stored in a cool and dry place before uses.

2.2. Formulation of composites

The formulation of untreated and treated WPC with specific ratios was summarised in Table 1. All the composites were carefully

prepared under the same processing condition as follows: the required amount of PE for each batch was firstly placed in a Brabender Plastograph twin-screw mixer and allowed to melt at 100 rpm and 190 °C for 2 min, and subsequently mixed with wood flour for 3 min. The lubricants and/or coupling agents were thus added into system and mixed for another 10 min. The resulted mixture was thus ground to pellets by using a Retsch cutting mill (SM 100, Germany). The ground blends were compression moulded on an electrically heated hydraulic press. Hot-press procedures involved 20 min preheating at 190 °C with no load applied followed by 10 min compressing at the same temperature under the pressure of 9.81 MPa, and subsequently air cooling under load until the mould reached 40 °C.

2.3. Characterisation of WPC microstructure

All the composites were transversely cut by using a sliding microtome with the nominal thickness of around 25 microns for the morphological investigation of the cross sections. The observation was conducted on a Leo 1430VP Scanning Electron Microscope (SEM) operating at 10 kV, all the samples were conductively plated with gold by sputtering for 45 s before imaging.

2.4. Tensile property analysis

Tensile properties of the composites were determined at a crosshead speed of 1 mm/min according to the standard BS EN ISO 527-2:2012 on an Instron 5900 testing machine with 30 kN load capacity. For each sample, the tensile property reported is the average of six measurements. The tensile properties of recycled PE were also measured and given for reference: tensile stress at maximum load 23.05 ± 0.17 MPa, tensile strain at maximum load 10.35 ± 0.32%, tensile modulus 2385.41 ± 133.25 MPa.

2.5. Dynamic mechanical analysis (DMA)

Dynamic mechanical properties of the composites were measured by using a dynamic mechanical analyser (Q800, TA Instruments, New Castle, USA) under single cantilever strain-controlled mode. The temperature ranges from −100 °C to 120 °C with a heating rate of 3 °C/min. The oscillation amplitude was 20 μm, the frequency was 1 Hz, and the specimen dimension was 17.5 mm × 10.8 mm × 1.4 mm.

2.6. Nanoindentation analysis

The samples for nanoindentation determination were prepared as follows: a sloping apex (around 45°) was created on the cross section of the sample by using a sliding microtome, thus the sample was mounted onto an ultramicrotome (Leica EM UC7, Germany) and transversely cut with a glass knife and a diamond knife to obtain an exceptionally smooth and flat surface. The cross section of the samples was firstly observed under an optical microscope to

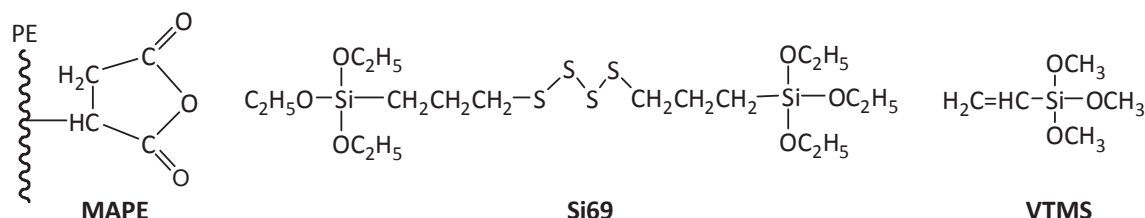


Fig. 1. Chemical formulae of the coupling agents.

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