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Non-destructive estimation of modulus of elasticity of wood polymer composite

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Abstract Dynamic modulus of elasticity (DMoE) of wood polymer composites, prepared with varying proportion of wood content, was determined using flexural vibration and ultrasonic pulse transit time methods. The DMoE was compared with the flexural modulus as measured by standard three point bending test. The elastic modulus of the composite increased with the increasing fiber content. Both vibration methods exhibited a strong linear association with static modulus. DMoE by ultrasonic pulse method was higher compared to flexural vibration method at low wood content and the difference diminished at 50% wood in composites. Both the dynamic modulus were higher as compared to the static modulus at all fiber loadings, however the difference between dynamic and static modulus reduced non-linearly with increasing wood content.

Keywords Elastic modulus · Flexural vibration · Non-destructive testing · Ultrasonic · WPC

Introduction

Non-destructive vibration methods are extensively used to determine elastic constants, damping and attenuation characteristics, fatigue behaviour, presence of flaws and other defects in the materials (Kolsky 1963; Bucur 1996; Tanasoiu et al. 2002; Hearmon 1966). Vibration methods require a very small force for a very short duration resulting in very small displacements. Thus such methods becomes more relevant in studying properties of visco-elastic polymeric materials where prolonged elongation, stresses and fatigue are may result changes in the structure of polymeric material and, consequently, in the properties of materials during static tests (Perepechko 1975). These changes are the results of orientation and other effects due to the high value of the mechanical stresses appearing in the material and the relatively large strains.

Polymers like polypropylene, polyethylene (HDPE and LDPE), elastomers, etc., are highly visco-elastic in nature and exhibit non-linearity in the stress–strain curves at a very small load. This non-linearity at small loads itself leads to ambiguity in determination of elastic modulus by static methods in such polymers (Tucker et al. 1998). Aggarwal (2008) observed significant variation in elastic modulus of HDPE, which is highly visco-elastic polymer, with change in the upper value of stress for determination of slope of stress–stain curve, whereas this variation was negligible in case of polystyrene which is a stiff polymer. In addition, the process parameters and formulations like fiber content, coupling agent and other additives strongly influences the properties of wood polymer composites (WPC). Since WPCs are now increasingly being used for semi-structural applications, they become prone to moisture absorption and fungal degradation particularly at high wood content. This may change their strength and stiffness over a period of time. Therefore, fast and reliable methods to assess such changes are necessary.

Vibration methods provide scope for quick assessment of important properties like Young's modulus and shear modulus, and such methods are relatively immune to the deficiencies associated with the static tests. Localizing resonance frequency of different vibration modes in a specimen of a specific shape and, measuring the speed and attenuation of a stress wave in the material are the two fundamental vibration methods. Tucker et al. (1998) used

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longitudinal vibration and ultrasonic method to determine modulus of elasticity and advocated the application of such methods to monitor quality of wood polymer composites. Orthotropic viscoelastic stiffness of cellulose fibre reinforced composites were measured using ultrasonic velocity measurements by Newell et al. (1997). Chauhan et al. (2006, 2009) used resonance frequencies of flexure and anti-flexure modes of vibration of disc shaped specimens in determining the dynamic elastic constants of WPC. Aggarwal (2008) used longitudinal and flexural vibration modes for estimating the dynamic modulus of elasticity of Wood-HDPE and Wood-Polystyrene composites and observed a strong correlation between static and dynamic modulus. However the resonance frequency methods require a well defined boundary condition for exciting specific vibration modes and may not be suitable for structures and fixed components. Nzokou et al. (2006) advocated that the confidence of WPC for structural uses will increase if the elasticity and strength of WPCs can be estimated non-destructively with high accuracy by applying a small deformation or vibration. In such case, pulse-transit time methods are generally immune to shape and size of the structure and can easily be adopted for in situ assessment of dynamic modulus and defects in composites. However, the speed of stress wave depends on the frequency of the wave and the modulus determined by this method is generally found to be higher than the static MoE and DMoE by resonance methods (Chauhan and Sethy 2016). The differences in dynamic MoEs determined by different vibration methods and their deviation from static MoE needs to be understood with varying wood content to ascertain the suitability of such vibration techniques for WPCs.

In this study, ultrasonic pulse propagation and flexural vibration methods have been tested for their efficacy in determining the modulus of elasticity of the composites at different filler level. The results are compared with the static test method. The modulus of elasticity of the composites at different wood content was also described based on the theoretic model.

Materials and methodology

Preparation of composites

Wood Polymer Composite were prepared by blending Rubber wood flour and polypropylene (Reliance Make H110 MA grade) at varying wood content (10–50% on weight basis) using a twin screw co-rotating extruder. The composites were prepared using in-house synthesized m-TMI-grafted-PP coupling agents (four coupling agents) having different grafting levels and molecular weights (Kale et al.

2016). Composites were also prepared without coupling agent. The composite granules were injection moulded into 127 mm × 12.3 mm × 6.6 mm ($l × w × t$) bar specimens which is used for determining flexural strength and modulus as per the ASTM 790. The same specimens were used for non-destructive testing. After injection moulding, specimens were conditioned for at least 24 h in the atmospheric conditions before testing. Five samples per formulation were selected randomly for testing. Each selected sample was weighed to an accuracy of 0.001 gm and measured for its volume to an accuracy of 0.01 cm^3. Density of the composite samples was determined by weight and volume measurements.

Testing of composites

Flexural vibration method

A resonance based vibration system designed at IWST Bengaluru was used to identify resonance frequencies of the flexural vibrations. To generate the fundamental mode of flexural vibration, the sample was suspended on fishing thread at the nodal points of the fundamental vibrations (0.224 × length) to create complete free–free boundary conditions. The suspended sample was gently tapped laterally from above at the centre point using a light hammer (specifically fabricated using a small metal ball) in such a manner that force was enough to excite the vibrational modes and yet was gentle enough not to give much momentum that can displace the specimen. A microphone was placed in the close proximity of the specimen to capture the vibration signals. The schematic arrangement of the experiment is shown in the Fig. 1. The Fast-Fourier transformation algorithm in-built in the software converted the time domain vibration spectrum into frequency domain spectrum with resonance frequency of fundamental mode and higher overtones.

The fundamental frequency of flexural vibrations was used to determine DMoE using the following equation (Chauhan and Sethy 2016)

$$DMoE = \frac{0.946 \times f^2 l^4}{t^2} \times \rho \qquad (1)$$
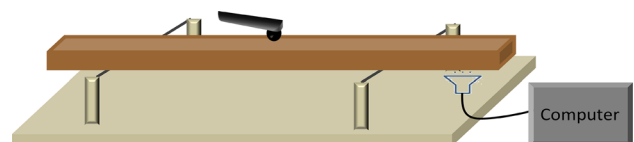

Fig. 1 Experimental set-up of the fundamental frequency of flexural vibration

where f is the fundamental frequency of flexural vibration, l is the sample length, t is the sample thickness and ρ is the density of the composite material.

Ultrasonic method

Ultrasonic method requires the placement of two piezo-electric transducers in contact with the ends, one transducer serves the ultrasonic pulse generator of very short duration which travels in the composite and the arrival of the pulse is detected by the transducer at the other end (Fig. 2). The transit time of the pulse is recorded over a distance and ultrasonic velocity in the material is determined. In this study, FAKOPP make ultrasonic timer tool was used. It has an ultrasonic transducer generating spike wave guide of 90 kHz frequency. Instrument is powered by 9 V batteries. A short ultrasonic impulse is generated by electronic excitation of the transducer. The pulse propagates in the material, and the arrival of pulse is picked-up by the second transducer. The signal at the second (receiver) transducer stops the timer, when reaches to the threshold level (0.12 V). The system has spiked probes enabling measurements in young trees, seedlings and in fixed structures. The time of pulse travel in the specimens of composites was in the range between 65 and 85 μs. With this time of travel and the known distance separating the transducers, i.e. the length of the specimen (nominally 127 mm), the velocity of the stress pulse was calculated using Eq. 2. The true transit time of the pulse was determined by subtracting time correction factor of 15 μs which is the transit time of pulse within the probes.

$$V = l/(t - 15) \tag{2}$$

where, V is the velocity, l is length of the sample and t is transit time. DMoE is calculated using Eq. 3

$$\mathrm{DMoE} = \rho \times V^2 \tag{3}$$

ρ is the density of the composite specimen.

Static testing

Flexural modulus was determined, on the same samples used for vibration testing, using a Shimadzu make universal testing machine (model AGIS10, 10 KN) as per ASTM D790-92 with a support span of 100 mm and a
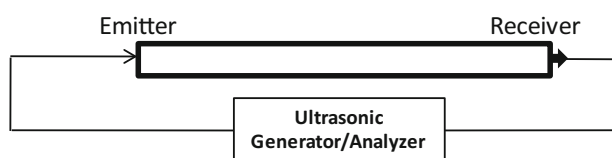

Fig. 2 Ultrasonic longitudinal test set-up

crosshead speed of 2.8 mm/min. The stress range for MoE determination was taken from 20 to 60 N irrespective of formulations. The flexural modulus was determined using Eq. 4

$$MoE = \frac{l^3}{4bd^3} \times \frac{\Delta P}{\Delta D} \tag{4}$$

where l is span length, b is sample width, d is thickness, ΔP is load difference and ΔD is difference in deflection at the centre point between the pre-defined loads.

Results and discussion

The injection moulded test bar specimens of the composite prepared at varying fibre content with m-TMI-g-PP coupling agent were tested for flexural vibration, ultrasonic transit time followed by three point bending test. Density of the composite material and modulus values increased with increasing filler content in all formulations irrespective of the coupling agent. To get the trend in modulus with fibre content, values of the density, dynamic modulus by flexural vibration and ultrasonic method and corresponding static flexural modulus at different fibre content for m-TMI-g-PP coupled composites is presented in Table 1.

It is evident that with increasing fiber content, all the measured parameters exhibited an increasing trend. The density of the injection moulded composite material increased from 0.903 g/cm^3 for virgin polymer to 1.075 g/cm^3 at 50% wood content representing about 19% increase in density. Whereas, static modulus by flexural method exhibited nearly 3.5 folds increase from virgin polymer to 50% wood composite. The dynamic modulus also exhibited very similar trend. This was as per the expectations as the modulus of the composite depends on the volume fraction of fibre and matrix polymer, and their respective elastic modulus (Chauhan et al. 2006). Since wood is high modulus material compared to polypropylene, the modulus of composite increased with increasing wood content. The modulus of the composites has been described based on several theoretical models. Rule of mixtures (ROM) and inverse Rule of Mixtures (IROM) have been reported to provide the upper-bound and lower bound of the modulus respectively (Mache 2015). The ROM and the IROM are defined as follows

$$E_C = V_f E_f + V_m E_m \tag{5}$$

$$\frac{1}{E_c} = \frac{V_f}{E_f} + \frac{V_m}{E_m} \tag{6}$$

Correspondingly a modified RoM can be obtained considering both RoM and IROM in the following form:

Table 1 Density, dynamic modulus and static modulus of WPC with m-TMI-g-PP coupling agent at different fibre content

Wood content (%)	Density (g/cm^3)	DMoE by flexural vibration (GPa)	DMoE by ultrasonic method (GPa)	Flexural modulus by static method (GPa)
0	0.903 (0.006)	2.17 (0.06)	2.81 (0.09)	1.05 (0.10)
10	0.917 (0.003)	2.84 (0.17)	3.32 (0.05)	1.43 (0.11)
20	0.955 (0.014)	3.40 (0.21)	3.77 (0.06)	2.01 (0.04)
30	0.986 (0.010)	3.96 (0.09)	4.04 (0.10)	2.54 (0.04)
40	1.021 (0.003)	4.59 (0.08)	4.65 (0.05)	3.04 (0.03)
50	1.075 (0.001)	5.13 (0.04)	5.30 (0.11)	3.64 (0.03)

Value in the parenthesis is the standard deviation

$$E_c = \frac{1}{2}\left(V_f E_f + V_m E_m + \frac{E_f E_m}{V_f E_m + V_m E_f}\right) \quad (7)$$

The static and dynamic modulus was predicted using model (Eq. 7) at different wood content. The volume fraction of wood (V_f) in composites at different wood fiber loading was estimated using Eq. 8

$$V_f = \frac{\rho_m w_f}{\rho_f w_m + \rho_m w_f} \quad (8)$$

where ρ_f, ρ_m, w_f and w_m are the oven dry density of Rubberwood, density of polypropylene, weight of wood and weight of polypropylene respectively. The oven dry density of Rubberwood was 0.575 g/cm^3 and the density of polypropylene was 0.0903 g/cm^3. Consequently, the volume fraction of wood in 10, 20, 30, 40 and 50% wood filled composites was estimated to be 0.15, 0.28, 0.40, 0.51 and 0.61 respectively. The modulus of composites as determined by three different methods and predicted using the model equation is shown in Fig. 3.

The model predicted values were closely in agreement with the experimental results in case of static bending and flexural vibration methods. In case of ultrasonic method, the model predicted values were over-estimated at wood
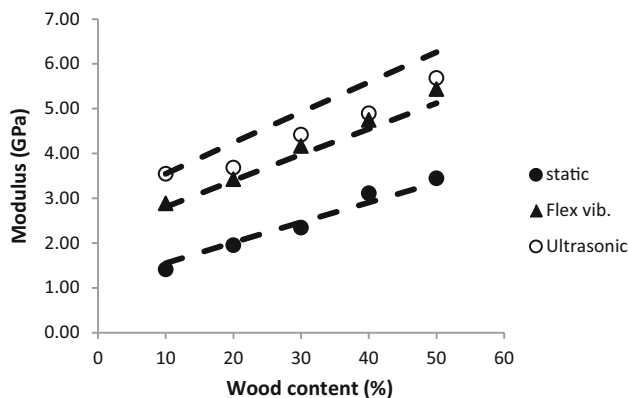
content of more that 20%. The accuracy of model depends on the value of modulus of individual components i.e. wood and polymer. The dynamic modulus measured by ultrasonic pulse transit-time in wood can vary significantly with the moisture content, sample length, grain angle, alignment of the probes. The accuracy of model in describing the modulus at different wood content, both static and dynamic (flexural vibration), clearly demonstrate the applicability of this model in predicting composite stiffness.

Dynamic modulus (determined by both the methods) was higher than the static modulus for all the specimens tested. This is mainly attributed to the difference in the straining rate in the dynamic and static methods, and creep phenomenon (Divos and Tanaka 2000). The loading rate in static test was 2.8 mm per minute while the resonance frequency of flexural vibration was in the range of 650 to 950 Hz and ultrasonic timer generate a pulse of 90 kHz suggesting significant variation in the straining rate in all three methods. The difference between DMoE by flexural vibration and static MoE is shown in Fig. 4 with respect to static MoE. There is a strong second order polynomial relationship between the difference and static MoE. It can be seen that dynamic modulus is nearly 120–140% higher
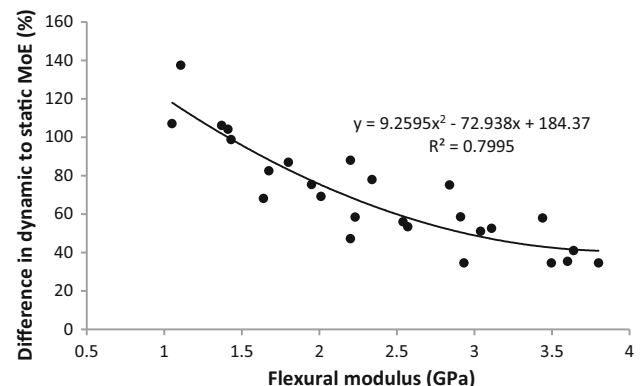

Fig. 3 Model predicted an experimentally observed modulus at varying wood content (symbols represent experimentally observed values and *dashed lines* represent model generated values)

Fig. 4 Difference in DMoE by flexural vibration and static MoE (%) with respect to static MoE

at the lower end of static MoE and around 35–40% higher at the higher end of static MoE. This is attributed to the change in the visco-elastic behaviour of composite material with increasing wood content. When wood filler is added to the matrix material, nature of material shifts from visco-elastic to elastic. Therefore the difference between the static and dynamic modulus reduced with increase in filler content. Aggarwal (2008) also reported smaller differences in dynamic and static MoE with increasing filler content in wood-HDPE composites. The difference was minimal in case of wood-polystyrene composite since polystyrene is highly stiff polymer.

To understand the relationship between dynamic modulus by flexural vibration and ultrasonic vibration, the dynamic modulus was measured in large number of samples (with and without coupling agent) and the relationship is shown in Fig. 5. There was a strong positive relationship between the two dynamic MoEs with a high coefficient of determination ($R^2 = 0.93$). The dynamic modulus by ultrasonic method was ranging from 2.7 GPa (for virgin polymer) to 6.0 GPa (for 50% wood filled composites). The modulus determined by flexural vibration was lower than by ultrasonic method in most of the cases except in few samples of 50% wood filled composites. The difference in dynamic modulus was depending of wood content in composites. In case of virgin polymer, the DMoE by ultrasonic was 29% higher than by flexural vibration whereas it was negligible for 50% wood filled composites.

The relationship of DMoEs with static MoE (Flexural modulus) is shown in the Fig. 6. It is clearly evident that DMoE as determined by either of the method is strongly related to the flexural modulus as determined by three-point bending test. However, the two trend lines tend to merge as the flexural modulus increased from 1 GPa (for virgin PP) to 3.60 GPa (for 50% filled composite). This implies that the difference between the two dynamic
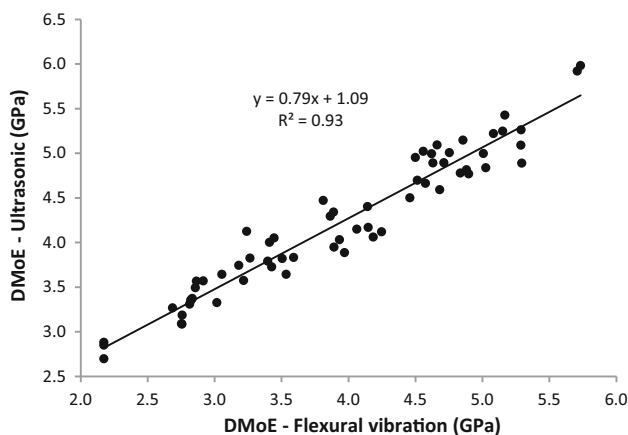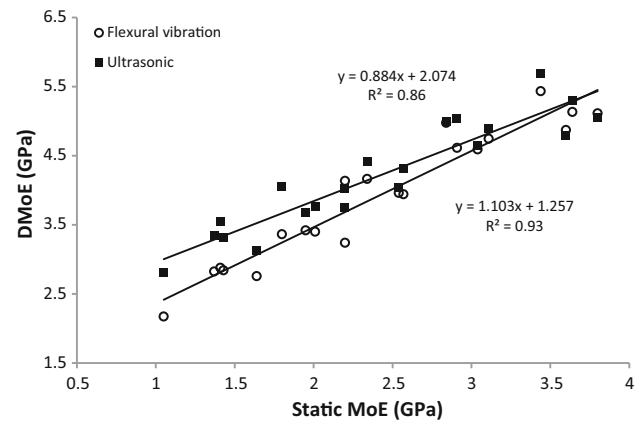

Fig. 6 Relationship of DMoEs with static MoE (flexural modulus)

modulus values diminishes with the increasing wood content in the composite material. The DMoE by flexural vibration exhibited a stronger relationship with flexural modulus (static) which may be attributed to the similarity in the load application in these two methods. In case of ultrasonic method, the pulse propagates along the length and generally is influenced by the fastest available path. Therefore, the pulse velocity may get affected by the presence of any defects (air pockets, mould compression etc.) during moulding. In contrast, the resonance method provides an average MoE of specimen (Chauhan et al. 2005).

Among the two vibration methods, flexural vibration method was found to be a better method over ultrasonics as the resonance frequency measurements were highly consistent and are immune to the impact point and impact force. In case of ultrasonic method, pin penetration in the sample and alignment of pins can result in variation in the transit time values. However, for assessment of structure or the component fixed in the structure, ultrasonic method is the suitable method. For quality control, flexural vibration method can effectively be used to assess stiffness or modulus of elasticity of the wood polymer composite material.

Conclusion

Both the non-destructive vibration methods (flexural vibration and ultrasonic pulse propagation) were found to exhibit a strong linear association with static modulus but the difference of dynamic to static modulus was found to vary with the fiber content in the composite. The strong association of static method with flexural vibration method suggest that the selection of stress points (20–60 N) on stress–strain curve during static testing was in the linear region for all formulation.

Fig. 5 Relationship between dynamic modulus by flexural vibration and ultrasonic vibration

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