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Errors in surface energy estimation of fibres for Liquid Composite Moulding processes and potential origins

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Contact

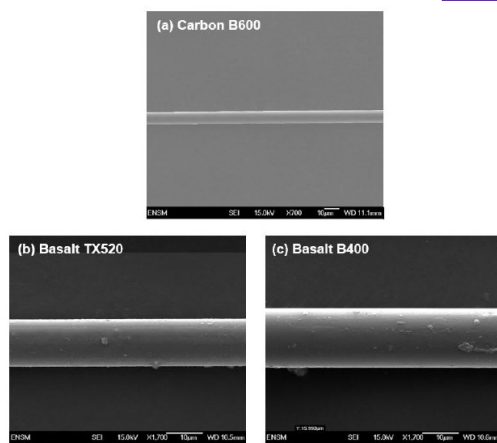
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Context

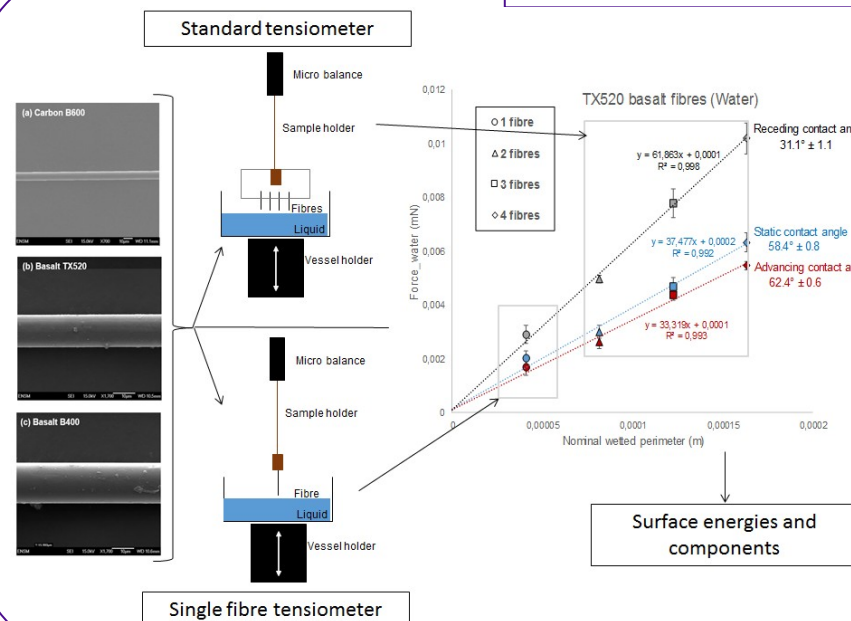
For a better understanding of the behaviour of new eco-reinforcements, such as recycled carbon fibres, during Liquid Composite Moulding processes (LCM), fibres surface analysis and wetting properties are studied. However, this type of analysis, using the Owens and Wendt relation requires special procedures, specifically for estimation of the contact angle. Based on two tensiometric methods, carbon and basalt fibres with different sizing are characterised in first approach. The main contribution of this study is to evaluate the error in surface energy and its components determination associated to the measurement of an alleged equilibrium contact angle deriving from static or quasi-static data.

Materials



Three types of fibre, presenting controlled morphologies were chosen to correlate tensiometric methods. Two types of basalt fibre having different types of sizing compatible with thermoset resins were tested. The first one, basalt fibres extracted from TX520 triaxial stitched fabrics provided by Basaltex are characterised by a silane-based sizing compatible with epoxy. The commercial designation is Basalt TX520. The second ones, basalt fibres extracted from B400 triaxial stitched fabrics provided by Basaltex have a sizing agent compatible with vinylester resin. Those fibres will be referred to as Basalt B400. Carbon fibre was also chosen because of its intensive use in composite materials made by LCM processes. Carbon fibres were extracted from Cbx600 fabrics (provided by Sicomin) with 12K yarns and a sizing agent compatible with epoxy. The areal weight is of $629 \pm 5\%$ g/m² and the nominal fibre diameter is of 6µm (13µm for basalt) with deviation lower than 0.5µm over 20 fibres.

Method



$$F_c = ma = p\gamma_L \cos\theta \quad (1)$$

$$\frac{\gamma_L (1 + \cos\theta_e)}{2\sqrt{\gamma_L^d}} = \sqrt{\gamma_S^p} \left(\frac{\sqrt{\gamma_L^p}}{\sqrt{\gamma_L^d}} \right) + \sqrt{\gamma_S^d} \quad (2)$$

A summary of the approach is shown in Fig.1. It is important to note that the aim of this study is not to compare equipment but to validate both methods or a crossover of the two methods [1-2]. To facilitate the experimental procedure, the basalt and carbon fibres, having relatively homogeneous and reproducible morphologies, were selected. Finally, the different contact angles, calculated from Wilhelmy equation (1), were used in the Owens and Wendt equation (2) [3] in order to evaluate the measurement errors in each type of fibre surface energy determination.

Results

Fibres	Liquids	θ_s / R	θ_a / R	θ_r / R
Basalt TX520	Water	58.4±0.8 / 0.992	62.4±0.6 / 0.993	31.1±1.1 / 0.998
	SP106	40.9±1.6 / 0.995	51.0±1.2 / 0.990	33.9±3.5 / 0.980
	Vinylester	Cos θ > 1 / 0.992	23.6±3.1 / 0.964	Cos θ > 1 / 0.983
Basalt B400	Water	63.7±1.4 / 0.969	68.8±0.9 / 0.954	41.8±0.7 / 0.984
	SP106	41.9±0.3 / 0.996	53.0±0.3 / 0.998	30.9±1.8 / 0.997
	Vinylester	23.8±4.8 / 0.972	41.8±3.8 / 0.955	Cos θ > 1 / 0.954
Carbon Cbx600	Water	61.6±2.0 / 0.975	69.8±1.1 / 0.977	42.1±1.5 / 0.988
	SP106	28.1±3.1 / 0.984	42.7±1.7 / 0.990	Cos θ > 1 / 0.988
	Vinylester	Cos θ > 1 / 0.983	Cos θ > 1 / 0.995	Cos θ > 1 / 0.967

Contact angle	γ_S^p (mN/m)	γ_S^d (mN/m)	γ_S (mN/m)	
Basalt B400	Static	20.54 ± 0.97	18.80 ± 0.12	39.34 ± 1.09
	Advancing	17.84 ± 0.51	16.67 ± 0.27	34.51 ± 0.78
	Receding	37.81 ± 0.25	17.36 ± 0.35	55.17 ± 0.60
Basalt TX520	Static	24.35 ± 0.55	18.89 ± 0.06	43.24 ± 0.61
	Advancing	22.14 ± 0.55	17.50 ± 0.15	39.64 ± 0.70
	Receding	45.97 ± 0.49	16.21 ± 0.22	62.18 ± 0.71
Carbon Cbx600	Static	18.41 ± 1.60	20.65 ± 0.61	39.06 ± 2.21
	Advancing	15.52 ± 0.90	20.59 ± 0.41	36.11 ± 1.31
	Receding	36.52 ± 1.19	18.95 ± 0.21	55.47 ± 1.40

Conclusions

Neither advancing nor receding quasi-static angles can be trusted to evaluate surface energies. Static contact angles have to be as close as possible to the actual equilibrium to assess reliable surface energies, especially on fibres