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

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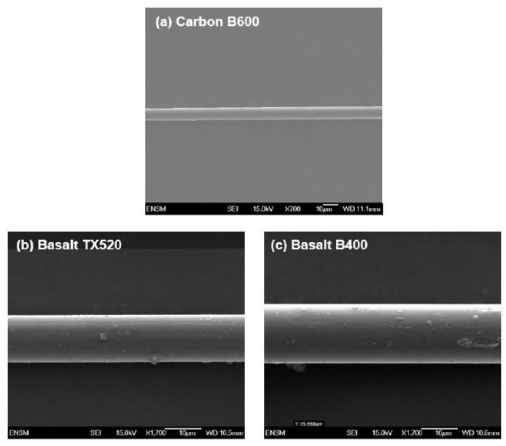
Context

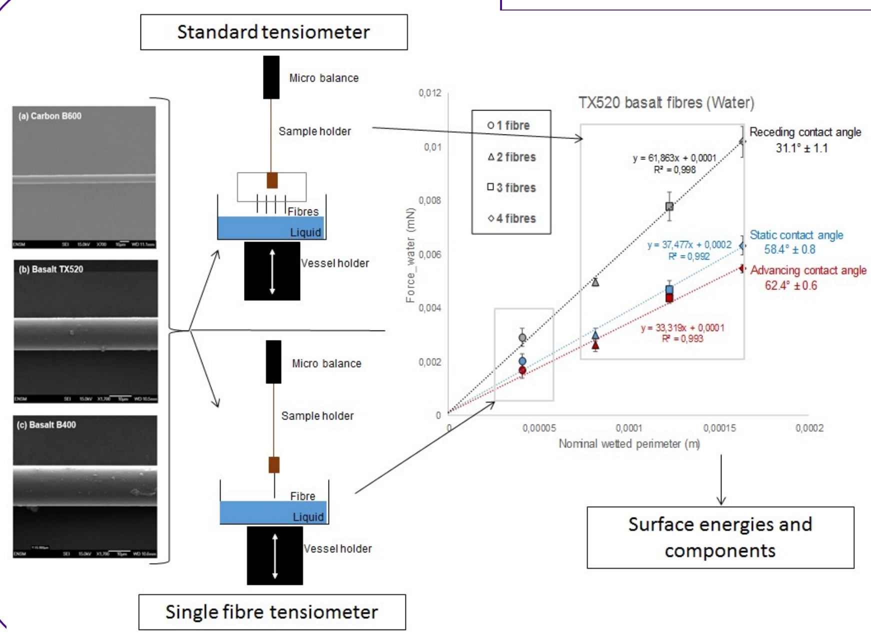
For a better understanding of the behaviour of new eco-reinforcements, such as recycled carbon fibres, during Liquid Composite Moulding processes (LCM), fibres surface analysis and wetting properties are studied. However, this type of analysis, using the Owens and Wendt relation requires special procedures, 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 ± 5% g/m² and the nominal fibre diameter is of 6µm (13µm for basalt) with deviation lower than 0.5µm over 20 fibres.

Method



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

$$\frac{\gamma_L\,(1+\cos\theta e)}{2\sqrt{\gamma_L^d}} = \sqrt{\gamma_S^p}\left(\frac{\sqrt{\gamma_L^p}}{\sqrt{\gamma_L^d}}\right) + \sqrt{\gamma_S^d} \qquad (2)$$

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	$\theta°_s$ / R	$\theta°_a$ / R	$\theta°_r$ / 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 receiding quasi-static angles can be trusted to evaluate surface energies. Static contact anglas haveto be as close as possible to the actual equilibrium to assess reliable surface energies, especially on fibres

References

[1] Pucci MF, Liotier PJ, Drapier S. Tensiometric method to reliably assess wetting properties of single fibers with resins: Validation on cellulosic reinforcements for composites. Colloids Surfaces A Physicochem Eng Asp 2017;512:26–33. https://doi.org/10.1016/j.colsurfa.2016.09.047.J. Apple, E. Berry and K. Current "The title of the conference paper". Proceedings of Conference Title, where it took place, Vol. 2, paper number, pp 2-22, 2002.

[2] Pucci, M. F., Liotier, P. J., Seveno, D., Fuentes, C., Van Vuure, A., & Drapier, S. (2017). Wetting and swelling property modifications of elementary flax fibres and their effects on the Liquid Composite Molding process. Composites Part A: Applied Science and Manufacturing, 97, 31-40.

[3] Owens DK, Wendt RC. Estimation of the surface free energy of polymers. J Appl Polym Sci 1969;13:1741–7. https://doi.org/10.1002/app.1969.070130815.

[4] Garat, W., Pucci, M. F., Leger, R., Govignon, Q., Berthet, F., Perrin, D., ... & Liotier, P. J. (2021). Surface energy determination of fibres for Liquid Composite Moulding processes: Method to estimate equilibrium contact angles from static and quasi-static data. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 611, 125787.

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