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IDENTIFICATION OF KEY PARAMETERS IN DYNAMIC WETTING AND DEWETTING PHENOMENA WITH MOLTEN POLYMERS FOR COMPOSITE PROCESSING

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Introduction

The main current research interest in the field of composite materials consists in minimizing the environmental impact of both materials and manufacturing. Having a good control of (bio)composite manufacturing implies a good knowledge of wetting phenomena occurring during impregnation of (natural) fibres by thermoset or thermoplastic polymers. It is known that capillary effects, and particularly capillary parameters like molten polymer surface tension and fibre surface energy, play an important role on impregnation. These effects coupled with the viscous ones are dominant during composite manufacturing [1,2]. The visco-capillary balance in dynamic wetting can be described by a hydrodynamic approach. In the literature, the de Gennes and Cox-Voinov theories [3] were used to fit experimental data (obtained mainly by optical methods) of liquid drops spreading on model substrates at standard conditions (room temperature). This procedure is not suitable in the case of fibres wetted by molten polymers for composite applications. Moreover, the influence of temperature and polymer molecular weight still remain unknown. The aim of this study was to set an experimental procedure that can be applied to fibres and polymers to investigate wetting dynamic and identify physical parameters such as a slip length, that can be inserted into numerical models to predict flow process [4].

The experimental procedure was validated on cellulosic films with different liquids. Paraffin oils are totally wetting liquids and they were used in order to evaluate the influence of temperature on wetting dynamics. Polyethylene glycols (PEGs) are partially wetting liquids; they were used to evaluate the effect of molecular weights (*Mn*) on wetting dynamics of molten polymers. An independent characterisation of the solid and of the liquids was carried out as well as solid/liquid dynamic wetting tests. Hydrodynamic models were used to fit the dynamic contact angle data. The results are meaningful for a better understanding of the influence of temperature, viscosity and liquid molecular weight on the dynamic contact angles and on capillary phenomena existing during the impregnation of fibrous reinforcements in composite processing.

Materials and experimental methods

Cellulosic films, referenced as NatureFlex™ 23 NP and gracefully provided by Innovia Films, were used in this study. Films have a thickness of 23 µm and are manufactured from biodegradable cellulose, which is derived from wood pulp. In order to consider cellulosic films as model substrates, such as ideal smooth and chemically homogeneous surfaces, morphology and surface chemistry of films were analysed by atomic force microscopy (AFM), Fourier-transform infrared spectroscopy (FTIR) and Wide-angle X-ray diffraction (WAXD).

Liquids used in this study were two paraffin oils, named (1) and (2) and three PEGs with *Mn* = 1450, 3350 and 10000 g/mol. Rheological tests were carried out with a Physica MCR 501 rheometer from Anton Paar to measure viscosity as a function of temperature. A Krüss DSA100 tensiometer equipped with a thermalized syringe and chamber was used to measure surface tension as a function of temperature *via* the pendant drop method (Figure 1-left) and density *via* the sessile drop method.

A Krüss K100SF tensiometer with a temperature controller was used to perform dynamic wetting tests between the cellulosic films and all the liquids (Figure 1-right). Regarding paraffin oils, three temperatures were considered for dynamic tests: 25, 50 and 75°C. Dynamic wetting tests of PEGs were carried out at T=75°C. Different velocities were imposed on the films for liquid immersion

(wetting) and withdrawal (dewetting). This protocol produced dynamic contact angles for a large range of capillary numbers *Ca*.

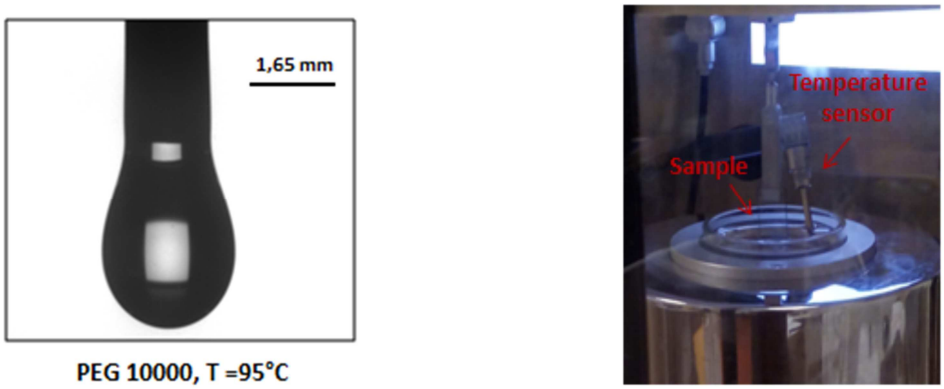

Figure 1: *Left: Pendant drop capture for surface tension measurement. Right: Immersion of a cellulosic film in a molten polymer at the K100SF tensiometer.*

Results and discussion

It was found that hydrodynamic models fit quite well experimental results of advancing contact angles (θ_a) for paraffin oils at different temperatures as a function of capillary number above Ca ≈ 10^{-3} (Figure 2). Varying the temperature modifies both surface tension and viscosity, changing the capillary number regime. This temperature variation does not remarkably affect the slip length, which is therefore a characteristic of the liquid in this hydrodynamic approach. Furthermore, the experimental data follow a same trend at various temperatures. For molten PEG at T=75°C, different trends were obtained for different *Mn*. This result suggests that the slip length depends on the chain length, which in turn affects the dynamic wetting. These points will be detailed in the communication.

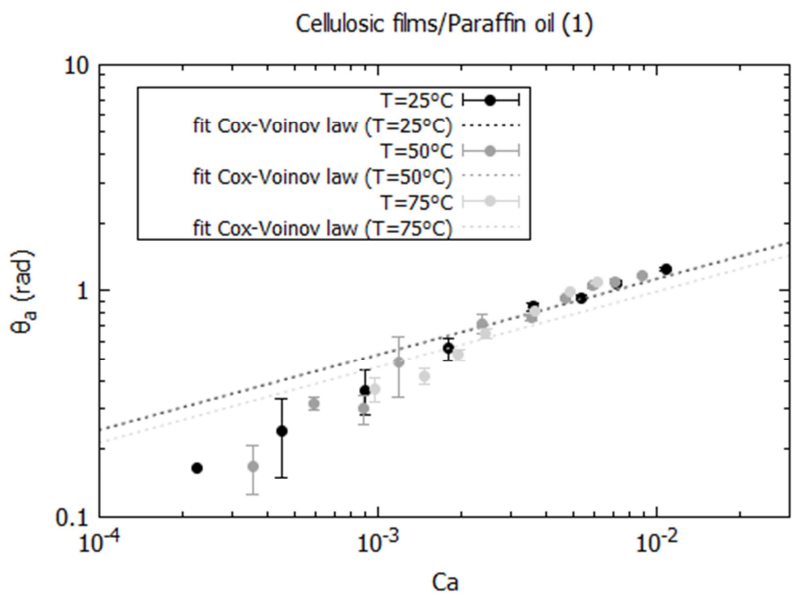

Figure 2: *Advancing contact angles as a function of capillary number and fits according to the Cox-Voinov law.*

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