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# RESIN CHARACTERISATION FOR NUMERICAL MODELLING OF THROUGH-THICKNESS RESIN FLOW DURING OOA PROCESSING OF THICK-SECTION WIND OR TIDAL TURBINE BLADES

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## ABSTRACT

The characterisation of a powder-epoxy resin has been carried out using differential scanning calorimetry (DSC) and parallel-plate rheometry, for both isothermal and dynamic conditions. The powder-epoxy has been identified as a candidate material for the cost-effective manufacture of thick composite sections in marine renewable energy (MRE) applications. The objective of this research was to investigate the powder-epoxy under different processing conditions, with the overall aim of developing a numerical resin flow model that can optimise material processing and ensure that high quality manufacturing is achieved. The experimental sets of data for DSC and rheometry have been fitted to a cure kinetics model and a chemorheological model, respectively. The cure kinetics model was implemented as a predictive tool in the chemorheological model in order to simulate the change in viscosity due to curing. Good initial results were achieved for both models; however, shortcomings in the experimental and theoretical methodologies were identified and outlined, and will be accounted for in future work.

## 1 INTRODUCTION

In recent years, the threat of global warming has become an ever-increasing issue within the public eye. Since the adoption of the Kyoto Protocol, many countries have set out new policies and targets to offset the current dependency on fossil fuels, and thereby reduce carbon emissions. A large portion of these policies and targets are based on improving the share of renewable energy within a country's total energy consumption. This is particularly true of OECD countries, as shown by the IEA/IRENA Joint Policies and Measures database for renewable energy [1], and is reflected in the EU's 2020 renewable energy targets.

As part of this drive towards cleaner energy production, researchers and policy-makers have identified the potential of marine renewable energy (MRE) as a self-sustaining, indigenous energy resource. In particular, MRE is attractive to countries with large coastal areas and sufficient resources in wave, tidal and offshore wind energy.

Recent assessments in Ireland [OREDP] have found that it would be possible to achieve high-scenario 2030 targets of 4.5 GW from offshore wind, and 1.5 GW from wave and tidal resources with a low risk of significant adverse effects to the environment; however, these figures are subject to commercial and technical feasibility [2]. In reality, to fulfil this potential, significant steps need to be made in both improving the quality of the current technology, and greatly reducing the cost of MRE devices.

Materials are an important area of research for MRE device manufacturers, due to both design and cost considerations [3]. Like wind turbines, most tidal turbine devices currently use fibre-reinforced polymers (FRPs) for their blades, because of their mouldability, resistance to corrosion in the harsh marine environment, and the significant weight savings which can be advantageous during installation and operation. Many wave energy converters are still being made from steel; however, companies such as Aquamarine Power have expressed interest in using FRP for future devices [4], most likely for the advantages mentioned above. Despite these advantages, MRE is also posing many new problems for the composites industry:

- The processing of thick sections is a major focal point due to the ever increasing size of components such as turbine blades (> 100 mm thickness for blade roots)
- Similar to the bigger onshore turbines, offshore wind turbines are so large now that they require the integration of carbon-fibre spars and carbon-glass transitions at the roots of blades
- Tidal turbine blades are much shorter than their wind equivalents, but still experience similar forces and moments, owing to the higher density of water (800 times that of air). This results in more rapid transitions in the blade profile, meaning greater angles of twist and more rapid ply drop-offs
- Most MRE devices must remain in-situ for periods of up to 20 years with little or no maintenance, withstanding fatigue loading in immersed, or near-immersed conditions
- Long-term material properties must be measured in seawater immersion and possibly under pressurised conditions (up to 4.0 bar at 30m depth, for example)
- Sea-mounted tidal devices can experience erosion due to sand and small stones damaging the blades

Aside from these challenges, the question that the MRE industry is really asking is, can our devices be made at a lower cost without compromising the quality? Installation costs of MRE devices are a major barrier to the technology and cost savings are targeted wherever possible. One of the targeted areas is materials and manufacturing [3], and yet components must be of a high enough quality to withstand all the conditions that are outlined above.

Increasingly, the aerospace industry has looked towards out-of-autoclave (OOA) materials and processes as a more cost-effective way of producing high quality composite components [5]. Vacuum-bag-only (VBO) prepregs are a type of OOA material that have been successfully used to create high-performance structures, and yet only require low-cost infrastructure and tooling [6]. Typically, in aerospace applications, VBO prepregs refer to carbon fibre beds that have been partially impregnated with a B-staged thermoset resin, but for MRE applications this can be extended to include glass fibres and basalt fibres as well. The VBO prepregs are normally laid-up and bagged on a moulding tool and heated within an oven, yet alternative processes exist that may offer even greater cost savings, such as the electrically-heated ceramic composite tooling developed by ÉireComposites Teo [7].

While these materials and processes offer great potential, it has been identified in other studies that OOA prepregs are sensitive to variation in key process parameters and can fail to meet the required quality if the correct conditions are not met [6]. As such, significant knowledge of candidate materials and processes is necessary to ensure that high quality components are achievable on a consistent basis.

The obvious first step towards acquiring this knowledge is characterisation of the candidate materials through advanced thermal and mechanical analysis techniques, such as differential scanning calorimetry (DSC), dynamic mechanical and thermal analysis (DMTA) and rheometry. Using information from these techniques, it is possible to develop an understanding of how the material behaves during consolidation and curing. Data from the experiments can be fitted to mathematical models to describe the cure kinetics and chemo-rheological behaviour of the polymer resin [8-11]. This has direct advantages for manufacturing whereby process parameters can be evaluated based on the values required for degree-of-cure and minimum viscosity.

These basic models can also be implemented in more advanced numerical models for simulation of resin flow. Resin flow models allow insight into the progression of the resin flow front during impregnation of the fibre-bed. These models are commonly based on Darcy's Law for flow of a fluid through a porous medium [12-14]. This type of numerical model can also be progressed further to incorporate heat transfer [14-16], thus providing a more comprehensive simulation of the actual consolidation and curing process. This form of modelling will be particularly useful for OOA techniques where heat is emitted from the surface of the tool and subsequently transferred through the composite plies. In this case, incorrect process parameters could lead to significant temperature differences within thick sections; preliminary testing has shown that such variation can significantly affect the quality of the finished component [17].

Combined with mechanical test data for both dry and immersed conditions, it should be possible to fully characterise candidate materials and determine their suitability for MRE applications. Furthermore, by using numerical modelling to identify an efficient processing window, the time and cost of manufacturing can be kept to a minimum. Development of a processing window, along with in-situ monitoring techniques, such as embedded fibre-optics, creates the potential to achieve significant cost savings while providing advanced quality assurance.

## 2 RESIN CHARACTERISATION

The current work is mainly focused on characterisation of a candidate resin which has been identified for its potential use in MRE applications. Data from the resin characterisation can be fitted to existing cure kinetic and chemo-rheological models, and subsequently used to predict the behaviour of the resin for any processing conditions.

### 2.1 Material

ÉireComposites Teo. has developed a glass-fibre reinforced material that is partially impregnated with a heat-activated powder-epoxy. This material has been used to manufacture 12.6 m wind turbine blades in a "one-shot" process with the heated ceramic tool previously mentioned [7]. As such, the powder-epoxy is suited for use in OOA processes and can be applied to fibre beds to form a sort of prepreg material, referred to as a "semi-preg". All reactants are already mixed in the powder and the curing reaction is heat-activated so that the "semi-preg" can be stored at ambient temperature. The resin manufacturer's recommended cure temperature is 180°C.

### 2.2 Cure Kinetics

Resin cure kinetics are commonly characterised using empirical models, expressed by rate equations for the degree-of-cure,  $\alpha$ . One auto-catalytic model that has been widely used is the Prout-Tompkins model [18], which has been successfully used for epoxies [9]:

$$\frac{d\alpha}{dt} = k[\alpha^m(1 - \alpha)^n] \quad (1)$$

where  $k$  is the temperature dependant kinetic constant, and  $m$  and  $n$  are the overall reaction orders. The temperature dependant kinetic constants are expressed by the Arrhenius equation:

$$k = A \exp\left(\frac{-E}{RT}\right) \quad (2)$$

where  $A$  is the pre-exponential constant,  $E$  is the activation energy,  $R$  is the universal gas constant, and  $T$  is absolute temperature.

In addition to the rate equation, the cure kinetics model can be improved by including an equation for the transition to a diffusion-controlled reaction [8, 10]. As cure progresses and the crosslink density increases, mobility of the reactants is reduced until a critical degree of cure is reached and further curing occurs through diffusion. Chern and Poehlein developed a diffusion factor [19] which can be added to Equation 1 to form a more accurate cure kinetics model, Equation 3.

$$\frac{d\alpha}{dt} = \frac{k[\alpha^m(1-\alpha)^n]}{1 + \exp[C(\alpha - \alpha_c)]} \quad (3)$$

where  $C$  is the diffusion control factor and  $\alpha_c$  is the critical degree-of-cure (the point at which cure becomes diffusion-controlled).

The model parameters are estimated by fitting experimental data to Equation 1; DSC is the most widely used technique for generating this data. DSC is used to measure the heat response of a resin sample undergoing the curing reaction, either for isothermal or dynamic conditions. The resulting exothermic peak in the measured heat response is a product of any curing that occurs. Thus, it is assumed that the rate of heat flow,  $dH/dt$  is proportional to the reaction rate,  $d\alpha/dt$ :

$$\frac{d\alpha}{dt} = \frac{1}{H_T} \frac{dH}{dt} \quad (4)$$

where  $H_T$  is the total heat of reaction. The degree-of-cure is calculated by integrating the heat response w.r.t time:

$$\alpha = \frac{1}{H_T} \int_0^t \left( \frac{dH}{dt} \right) dt \quad (5)$$

### 2.3 DSC testing

The powder-epoxy was tested using a Q100 DSC by TA Instruments. For all tests, 5-10 mg samples of powder-epoxy were weighed and hermetically sealed in aluminium pans. A series of isothermal and dynamic tests were used to accumulate pertinent data for cure kinetics analysis and modelling.

For isothermal testing, a temperature range from 150°C to 200°C was used to acquire a sufficient range of data either side of the recommended curing temperature (i.e. 180°C). This data can be used to determine the temperature dependence of the kinetic constants and reaction orders in Equation 1. To try and simulate isothermal conditions as closely as possible, all tests were rapidly ramped from -15°C to their final temperature at 30°C/min.

For dynamic testing, heating rates of 1.5 - 20°C/min were used, with tests starting at -15°C and finishing at 300°C. Results of the dynamic tests can be used to validate the model parameters estimated from the isothermal data [8].

### 2.4 Chemo-rheology

A number of empirical chemo-rheological models exist that can be used to express viscosity as a function of chemical conversion. One such model was developed by Castro and Macosko for polyurethane RIM systems [20], but has been widely used for other systems, including epoxy resin films [8, 11]. The Castro-Macosko model is expressed as;

$$\eta = \eta_0 \left[ \frac{\alpha_g}{\alpha_g - \alpha} \right]^{(A+B\alpha)} \quad (6)$$

where  $\eta_0$  is the temperature-dependant viscosity at zero degree of cure,  $\alpha_g$  is the degree of cure at gelation,  $\alpha$  is the degree of cure at a given time and temperature, and  $A$  and  $B$  are fitted parameters which can be temperature-dependant. The temperature-dependant viscosity,  $\eta_0$  can be expressed by the following relationship,

$$\eta_0 = A_\eta \exp\left(\frac{E_\eta}{RT}\right) \quad (7)$$

where  $A_\eta$  is the pre-exponential factor for initial viscosity,  $E_\eta$  is the viscosity activation energy,  $R$  is the universal gas constant, and  $T$  is the temperature in Kelvin. Equation 7 can also be expressed as a linear relationship between log viscosity, the universal gas constant and the inverse of temperature,

$$\ln \eta_0 = \ln A_\eta + \frac{E_\eta}{RT} \quad (8)$$

## 2.5 Parallel-plate rheometry testing

Rheometry was carried out using a HR-2 Discovery Hybrid Rheometer by TA Instruments. Plates with a diameter of 25 mm and a gap height of 1000 micron were used for testing in oscillation mode at an angular frequency of 10 rad/s. Powder-epoxy samples were loaded onto the plates using a melt ring to melt and compact the samples before trimming away excess resin. All tests were started at 70°C and were terminated after the gel point had been reached. As with the DSC testing, both isothermal and dynamic tests were used to investigate the behaviour of the resin for different cure conditions.

In order to satisfy isothermal conditions for the isothermal testing, an initial heating rate of 20°C/min was used to ramp up the test temperature as quickly as possible. Based on the DSC results, it was determined that 170°C was the maximum temperature that could be reached at 20°C/min before curing commenced. The chosen temperature range for testing was 150 - 170°C. Fitting isothermal data to Equation 5 allows for estimation of the unknown model parameters.

Dynamic testing was carried out with heating rates of 1.5 - 10°C/min. The initial pre-cure viscosity data attained through dynamic testing can be fitted to Equation 7 in order to determine the temperature-dependant viscosity at zero degree of cure.

## 3 RESULTS AND DISCUSSION

The data from testing was compiled and processed in MS Excel before being imported into MATLAB. Using the empirical models outlined in the previous section, it was possible to develop fitting parameters for the prediction of resin behaviour under varying process conditions. This section presents the predictions and discusses their accuracy and validity.

### 3.1 Cure Kinetics

For the cure kinetics model, fitting parameters were achieved using isothermal DSC testing. The raw data was taken from TA's Universal Analysis software. The heat flow data with respect to time (Figure 1) was the basis of the empirical model. It is apparent from Figure 1 that although the exothermic peaks were on the same scale as other epoxies [8, 9], the reaction was very rapid. At 200°C, curing had finished after 10 minutes. This meant that the powder-epoxy produced a very low overall exotherm; approximately 165-180 J/g, whereas epoxies typically produce 425-435 J/g [8-10]. Having such a small exotherm during the cure reaction would be very advantageous for manufacturing thick-section components because it reduces the risk of thermal runaway and subsequent thermal degradation of the component.

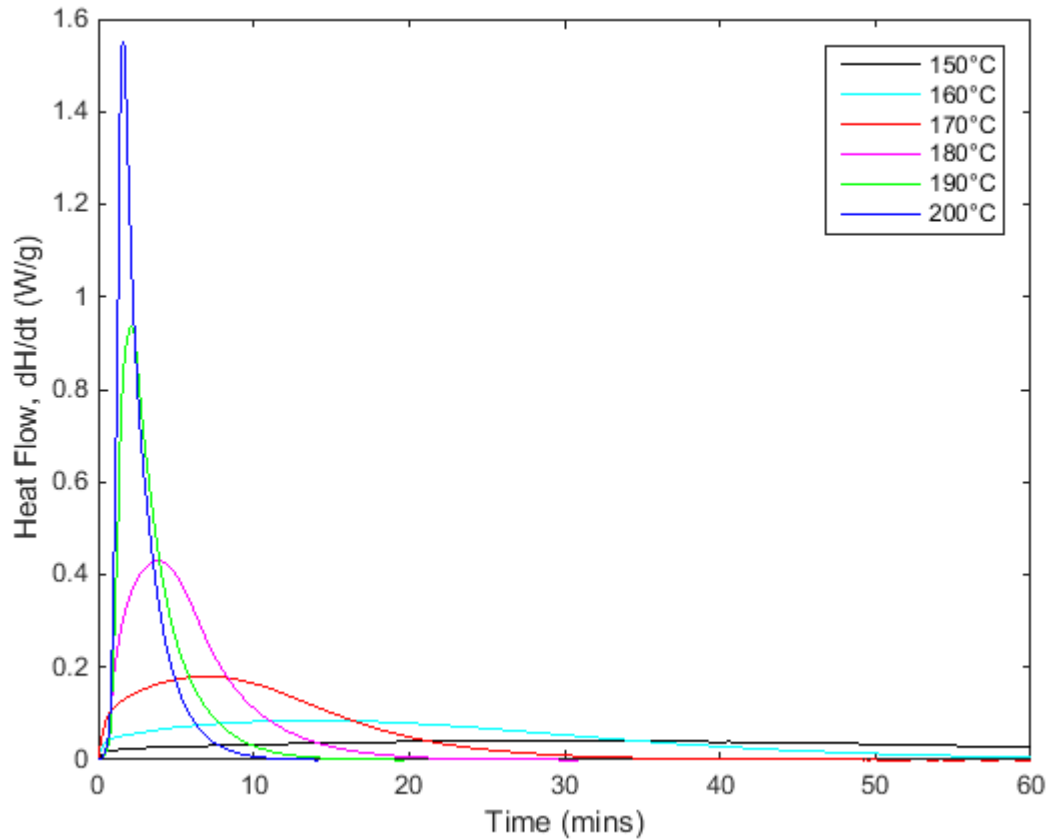


Figure 1: Raw data from isothermal DSC testing.

The above data was integrated in order to determine the degree-of-cure with respect to time, while Equation 4 was used to produce data for the reaction rate. MATLAB's in-built nonlinear least-squares curve fitting function was used to estimate all the fitting parameters for Equation 3. Variation was found in some of the parameters, which is believed to be a result of an inadequate number of test samples resulting in deviation from standardised test methodologies [21]. As such, linear fits were used to approximate the variation in the parameters with respect to temperature. Figure 2 compares the model predictions and experimental data for the degree-of-cure vs time. Although the model seemed to be relatively accurate until approximately 75% cure, there was significant variation in the test data above this point due to the discrepancies mentioned above. Furthermore, it was found that MATLAB's ordinary differential equation solver, ode15s, failed to compute the model for higher cure temperatures (i.e. 190°C and 200°C). It was thought that the equation may be too stiff for these temperatures due to the very rapid changes in reaction. Work will continue to try and resolve this problem; however, in reality, such rapid reactions are unlikely to be induced in manufacturing anyway because lower heating rates are typically used to achieve more uniform heating of a component and to avoid thermal runaway.

The dynamic test data was also processed and then imported into MATLAB for comparison with modelled data, the results of which are shown in Figure 3. Once again, the model prediction showed relative accuracy up until 75% cure which suggests that it has potential for development. For each heating rate, the model predicted a higher final degree-of-cure being achieved, but this may be due to the lower values of total heat flow that were measured for dynamic testing i.e. the average total heat flows for isothermal and dynamic testing were 180 J/g and 165 J/g, respectively. Further testing is needed to resolve these discrepancies.

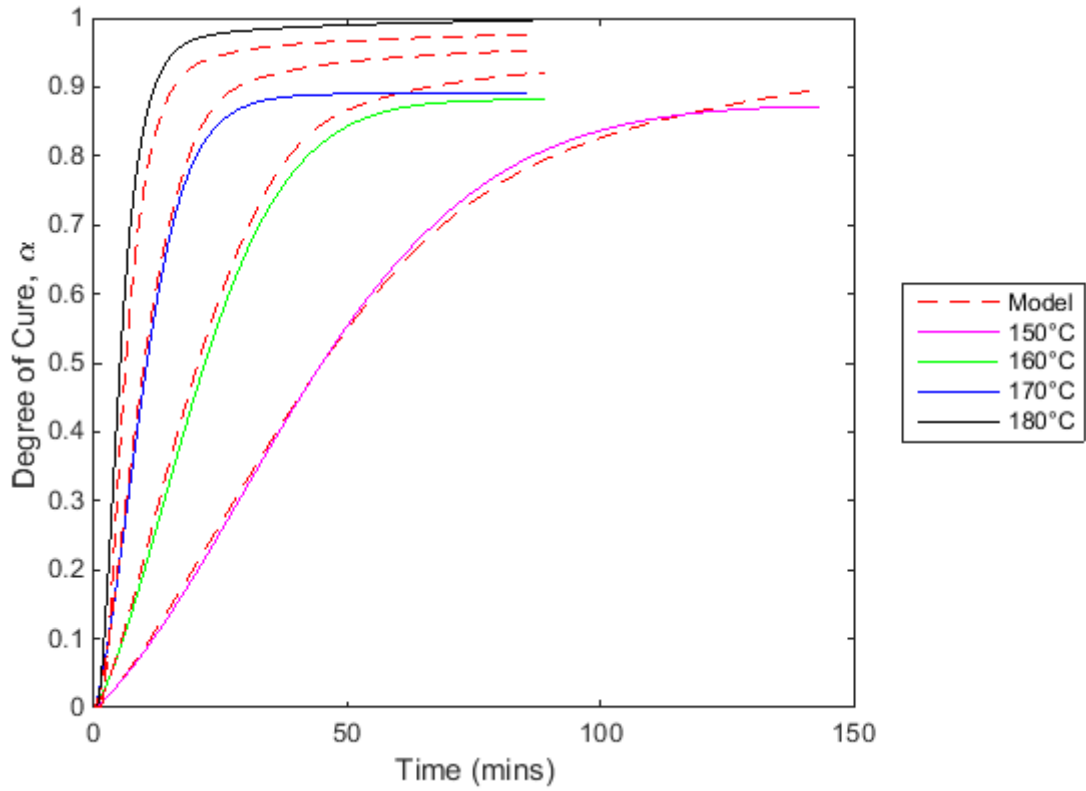


Figure 2: Comparison of model predictions and experimental data for isothermal DSC testing.

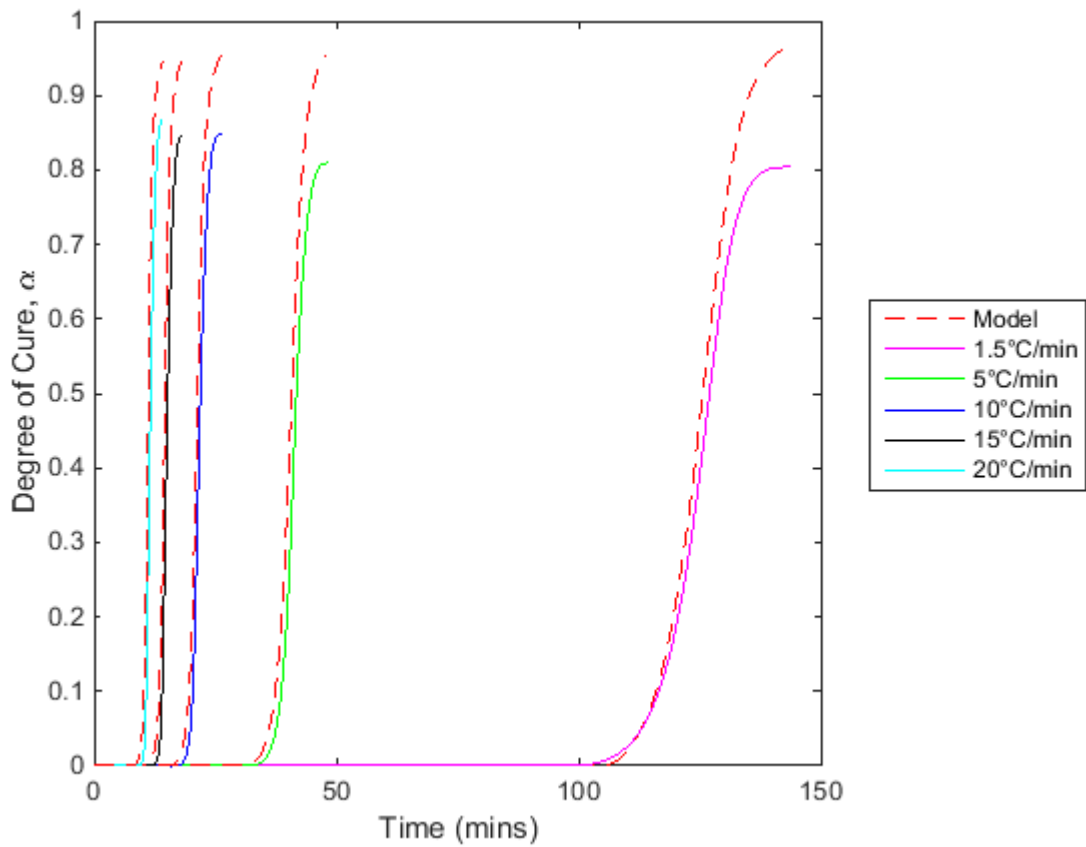


Figure 3: Comparison of model predictions and experimental data for dynamic DSC testing.



### 3.2 Chemo-rheology

The raw data from the parallel-plate rheometry showed that minimum viscosity values as low as 0.5 Pa.s were found with higher heating rates (i.e. 10°C/min). At the recommended heating rate of 1.5°C/min, a minimum viscosity of 4 Pa.s was recorded. This latter viscosity value is very competitive with what has been reported for similar OOA epoxy resins such as HexPly® M56 (Hexcel) and TC275-1 (TenCate). With a heating rate of 1°C/min, HexPly® M56 will reach a minimum viscosity of about 8 – 9 Pa.s, while TC275-1 can achieve 12 Pa.s at 3°F/min [22, 23]. Both of these resins are designed for VBO cure, which would suggest that the powder-epoxy is capable of providing a sufficiently low viscosity for this form of processing.

The time-temperature data for each rheometry test was inputted into the cure kinetics model to predict degree-of-cure with respect to time. Using these values in conjunction with the crossover point in storage and loss moduli, the gel point was estimated to occur at approximately 25% conversion. This was considered low for epoxy which is typically 45% or higher [8, 10, 24].

The linear relationship described by Equation 8 was populated with the zero-cure viscosity data from the dynamic tests. The pre-exponential constant,  $A_\eta$  and the activation energy,  $E_\eta$  were estimated to be  $6.914 \times 10^{-13}$  Pa.s and 99.25 kJ/mol, respectively. Using MATLAB's curve fitting toolbox, the experimental viscosity data and degree-of-cure predictions were fitted to the Castro-Macosko model (Equation 6) using a nonlinear least-squares method. Parameters  $A$  and  $B$  were found to have the following temperature dependencies;

$$A = 0.141 T - 53.28 \quad (9)$$

$$B = -0.844 T + 353.83 \quad (10)$$

The chemorheological model was run in MATLAB using the time-temperature data from each rheometry test and the corresponding cure kinetics model predictions. Figure 4 shows that the model predicted greater variation in the initial zero-cure viscosity than what was actually measured for isothermal conditions; this was magnified by showing viscosity on the log scale. Despite achieving an average R-squared value of 0.97 between the experimental data and the linear approximation for the zero-cure viscosity (Equation 8), there were obvious discrepancies between the two, as shown in Figure 5.

In terms of the model's performance, any poor fit at high viscosity was not considered to be a significant issue because there would be little chance of flow occurring at these values for VBO processes. On the other hand, minimum viscosity is highly important for any resin flow problems. In some cases, the model produced relatively accurate predictions in the lower viscosity range (within 1 – 2 Pa.s of the experimental result); however, the overall model fit at lower viscosities was slightly less accurate than expected.

As it stands, the linear assumption made in Equation 8 is questionable for the powder-epoxy when using the standard Castro-Macosko model. Khoun et al have shown that using a modified chemorheological model can produce accurate predictions for epoxies based on the use of two temperature dependent viscosity constants [10]. A similar approach may be implemented for modelling the powder-epoxy in future work, along with an increased number of test samples for each set of test conditions.

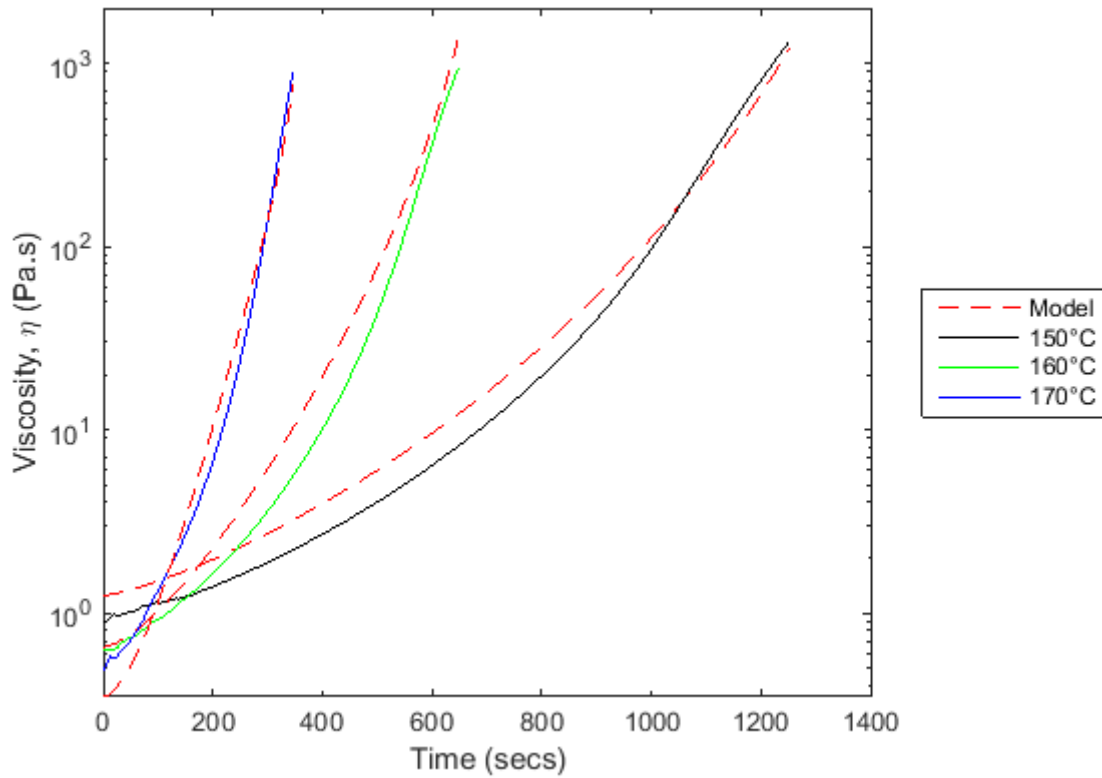


Figure 4: Comparison of model predictions and experimental data for isothermal rheometry testing.

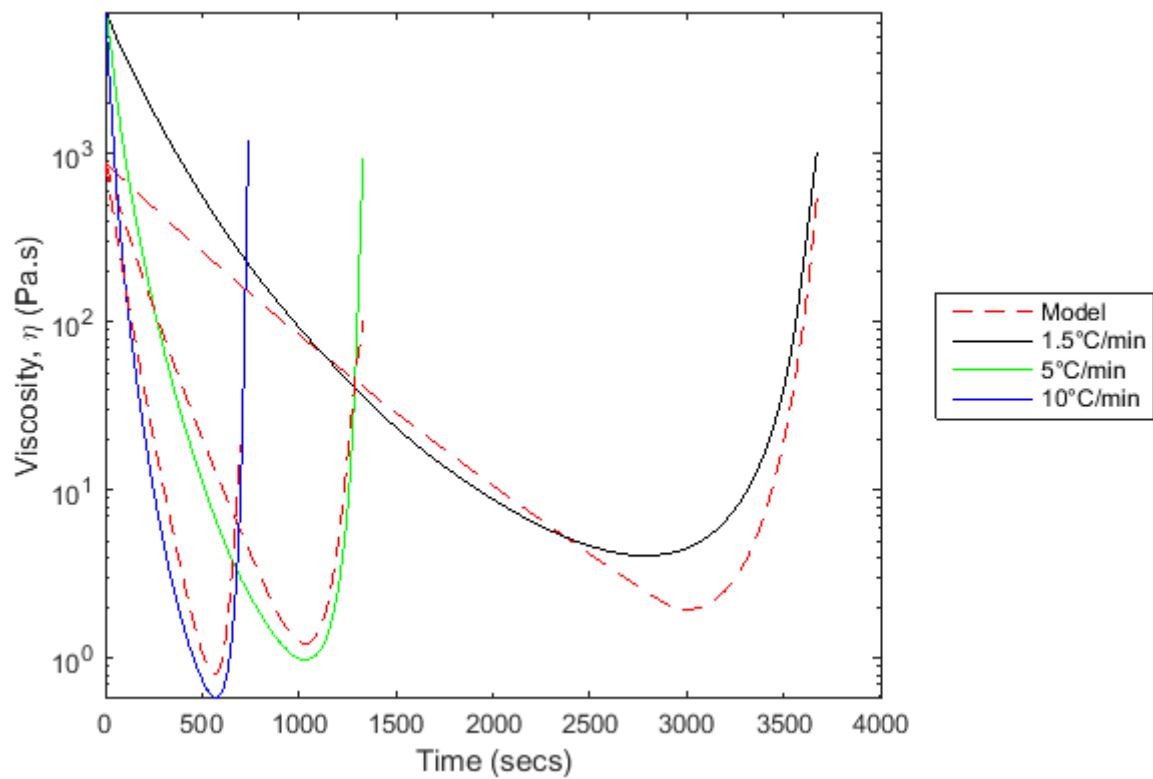


Figure 5: Comparison of model predictions and experimental data for dynamic rheometry testing. Note the discrepancies between the linear approximation for the zero-cure viscosity and the experimental data, particularly for 1.5°C/min.

## 4 CONCLUSIONS

The research presented the resin characterisation techniques and modelling methodology being used for the development of a numerical through-thickness resin flow model. The main body of the work was to improve knowledge of the powder-epoxy's behaviour under various processing conditions, and to use experimental data from DSC and rheometry to establish a chemorheological model that could accurately predict this behaviour based on time-temperature data. The initial results were positive and show that the powder-epoxy could provide an important development in OOA materials; in particular, for thick-section components manufactured using VBO processes.

DSC testing showed that the powder-epoxy is a very fast reacting resin that produces less than half the exotherm that is expected from standard epoxies. This is advantageous for manufacturing thick-section components such as those used in tidal turbine blades or wind turbine blade-spars, where 'thermal runaway' is a significant risk. It was possible to accurately model the material's reaction until 75% conversion for both isothermal and dynamic conditions. Variation in the experimental results meant that above 75% the model became significantly less accurate; however, this was attributed more to discrepancies in the two sets of data, rather than any major inaccuracy in the modelling technique. The model's accuracy should improve if a more complete and improved set of testing is carried out.

Results of the parallel-plate rheometry tests showed that the powder-epoxy exhibited relatively low minimum viscosity values for different isothermal and dynamic conditions. These values were shown to be very competitive with comparable OOA materials that were designed for VBO cure. As such, this would suggest that it is very well suited to VBO processes. By inputting the rheometry time-temperature data into the cure kinetics model, it was possible to estimate that gelation for the powder-epoxy occurs at a 25% conversion, which was lower than expected. Given that the cure kinetics model gives a relatively good fit at low degree-of-cure values, it is likely that this is an accurate estimate.

The cure kinetics model was also implemented within the chemorheological model so as to predict the change in viscosity due to an increasing degree-of-cure. It was found that the linear assumption for zero-cure viscosity returned a relatively good fit to the experimental data as a first approximation, but that an improved modelling approach would be required for greater accuracy.

Overall, the resin characterisation was beneficial in expanding processing knowledge of the powder-epoxy material, which is vital if it is to be considered as a high-quality, cost-effective alternative for MRE component manufacturing. For a first approximation, the relative accuracy of the chemorheological model suggests that there is sufficient potential with which to progress the research. Further testing and improved methodologies should generate better experimental data, and a revised modelling approach should build towards a validated predictive model.

## 5 FUTURE WORK

Under the research programme of the Science Foundation Ireland-funded MaREI Centre, the epoxy powder is being developed further for use in MRE applications with glass fibres and basalt fibres, as supplied by industrial partners. Likewise, the resin will be developed for use with carbon fibres as part of the MARINCOMP IAPP project.

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