Investigation of cure kinetic model and gel point for a vinylester-based SMC material

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Abstract. The capability of modeling the process of Sheet Moulding Compound by numerical tools requires detailed characterization of material in different conditions. A crucial aspect of this technology involves determining the required curing time and the onset of material gelation. These aspects are essential for controlling both the molding time and the flowability of the material. The purpose of this study is to analyze the kinetic and rheological properties of a commercial vinylester-based Sheet Moulding Compound material. The Kamal-Sourour model is employed to monitor the evolution of the Degree of Cure. The model parameters are determined by fitting Differential Scanning Calorimetry data using an ordinary least squares method and validated through a secondary analysis conducted under isothermal conditions. The findings demonstrate a robust correlation with the experimental data, with a deviation of less than 4%. The gel point is evaluated by comparing the conventional methodology employed with a rotational rheometer to an innovative approach utilizing a high-resolution camera able to monitor the flow of the material while compressed. The comparison of the outcomes indicates a discrepancy of approximately 3%, demonstrating the validity of this novel approach for determining the gel point.

Introduction

The increasing request for lightweight components in the automotive and transportation industries necessitates the adoption of advanced materials for both interior and exterior applications. Achieving an optimal balance between vehicle performance and weight reduction is crucial for minimizing fuel consumption while increasing vehicle safety [1]. Carbon Fiber Reinforced Plastics (CFRP) have emerged as the preferred solution across automotive, aerospace, and sporting goods sectors, owing to their exceptional specific properties. The carbon fiber composites market was valued at approximately USD 18.4 billion in 2021 and is forecasted to experience a compound annual growth rate of 6% from 2022 to 2030 [2]. To address this demand, it is essential to adopt advanced manufacturing technologies that enable the production of lightweight components, thereby improving manufacturing efficiency while maintaining high production flexibility [3]. In this context, Out-of-Autoclave methods, such as compression molding, offer notable savings in cost and time while maintaining design versatility and excellent performance properties [4]. Sheet Molding Compounds (SMC) materials are discontinuously reinforced polymer composites produced by compression molding. SMC composites typically consist of a thermoset resin reinforced with chopped carbon or glass fibers, generally grouped in randomly distributed bundles, and other additives and fillers, available in the form of pre-impregnated sheets. The sheets are cut and stacked to create an initial charge, which is subsequently molded onto a part through a compression molding process. Traditional trial-and-error methodologies are frequently employed in process optimization, requiring extensive testing campaigns to address the numerous factors that affect part performance. This approach is inherently inefficient, requiring considerable resources to evaluate each potential variable while generating a significant volume of waste [5]. An alternative method to optimizing the manufacturing process involves implementing numerical tools to simulate the SMC part production. To estimate the final properties of components, it is essential to predict potential defects using numerical modeling tools [6,7]. Indeed, the compression molding process may involve multiple phenomena, including heat transfer, thermosetting resin curing, fiber reorientation, fiber-matrix separation, weld line formation, mold friction, and void air release, all of which influence the final quality of the product [8,9]. Accurate definitions of aspects, such as cure kinetic behavior and the flowability of the material, are essential for achieving reliable and effective outcomes in research and application [10]. Commercial software utilizes analytical models to predict the evolution of the Degree of Cure (DoC) during the molding process and to characterize the viscous behavior of the material during flow. The first aspect is critical, as the duration the material remains in the mold directly influences production costs [11]. The viscosity of a material during processing is defined by models that require the identification of the Gel Point (GP) [12]. The GP represents the threshold at which the material transitions from a viscous state, capable of flowing, to an elastic state, where it ceases to flow. It directly influences the capability of the material to fill the mold and may contribute to the formation of unfilled regions during compression molding [13]. A conventional method for defining the GP of thermosetting resin involves utilizing a rotational rheometer to identify the crossover point between the Storage and the Loss modulus [14]. This analysis necessitated the use of testing samples with dimensions that were not excessively large. Indeed, for example, an increase in the thickness of the specimens can lead to the formation of an inhomogeneous temperature field in the stack direction. Moreover, an increase in the diameter typically leads to more stable data [15]. However, rheometers are generally constrained by the limited dimensions of their rotational plates.

Therefore, the aim of this study is to identify an alternative methodology, less dependent on these factors, for characterizing the GP of a Carbon Fiber SMC material. A high-resolution camera was employed to monitor the flow of an uncured sample positioned between two heated parallel plates. The Kamal-Sourour model is implemented for modeling the cure kinetics behavior of the material during tests, determining the values of DoC at which the GP is reached. The experimental results are compared with those obtained from a conventional analysis performed by rotational rheometer.

Material and Methods

Material. The material used in this work is STR120N131, a vinylester-based prepreg reinforced with carbon fiber, type TR50S, reinforced SMC produced by Mitsubishi Chemical Carbon Fibre and Composites GmbH. The nominal fiber volume fraction is 53%, and strands are randomly distributed (in-plane) in the 2 mm thick prepreg sheets.

Cure kinetic analysis. Differential Scanning Calorimetry (DSC) is a traditional analytical technique used to determine the kinetic parameters of the curing reaction by fitting experimental data to mathematical models. The crosslinking process is evaluated under dynamic conditions at heating rates of 2, 5, and 10 °C/min using a 214 Polyma DSC (NETZSCH-Gerätebau GmbH) with an intracooler in a nitrogen atmosphere at 60 ml/min. To ensure the reliability of the results, each DSC measurement is replicated three times. The prepreg material is prepared by cutting samples to an average weight of 15.7 ± 2.2 mg and then sealed in aluminum crucibles with pierced lids. Samples are heated from 20 °C to 250 °C, followed by a *burn-off cycle* characterized by an increase to 500 °C at 30 °C/min and an isothermal stage of one hour to achieve complete matrix degradation. After cooling, the samples are weighed to determine the specific resin mass content.

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The heat flow is monitored and normalized to the matrix fraction, and then the DoC is directly evaluated from it during the crosslinking reaction by Eq. 1:

$$\alpha\,(t) = \frac{\Delta H_t\,(t)}{\Delta H_{tot}} \qquad (1)$$

ΔH_t represents the enthalpy calculated from the beginning of the reaction to a specific time t, and ΔH_{tot} is the total enthalpy released during the cure. The DoC can vary between zero and one, where zero denotes an uncured state, and one indicates a fully cured state.

Reaction rate. Curing evolution models are generally based on differential equations, with the Kamal–Sourour model being one of the most prevalent analytical models employed to characterize the kinetic reaction [16]. The curing function merges an autocatalytic reaction term with a curing reaction of nth order (Eq. 2), and it is defined as:

$$\boldsymbol{\frac{d\alpha}{dt} = (k_0 + k_1\alpha^m)(1-\alpha)^n} \qquad (2)$$

In the model, $\frac{d\alpha}{dt}$ is the reaction rate, m and n represent the reaction orders, while k_0 and k_1 are described by Arrhenius equation (Eq. 3):

$$k_i = a_i \exp\left(-\frac{E_i}{R\,T}\right) \qquad (3)$$

For i = 0, 1, where a_i represents the fitted rate coefficient, E_i denotes the activation energy, R is the universal gas constant, and T is the curing temperature in °K. The six parameters are estimated by fitting the experimental DSC data using an Ordinary Least Squares (OLS) method that minimizes the following criterion (Eq. 4):

$$OLS = \sum \left[\frac{d\alpha}{dt}_{exp(t)} - \frac{d\alpha}{dt}_{model}(t)\right]^2 \qquad (4)$$

where $\frac{d\alpha}{dt}_{exp}$ is the experimental value and $\frac{d\alpha}{dt}_{model}$ is the prediction of the reaction rate by the Kamal-Sourour model.

Isothermal cycle analysis. A subsequent analysis, conducted in isothermal conditions, is performed to validate the outcomes of the Kamal-Sourour model by comparing the predicted and measured final DoC at three distinct temperature levels. A squared sample, approximately 40 mm on each side and composed of two stacked layers of SMC, is placed between two heated parallel steel plates, each with a dimension of 100 mm. The material is subjected to initial heating to target temperatures of 100°C, 120°C, and 140°C. Then the temperature is kept constant for one hour, after which the sample is cooled down. The evolution of the DoC can be monitored with the Kamal-Sourour model, using the temperature values registered during the test by a thermocouple positioned inside the material.

After the isothermal cycle, three DSC specimens are cut from each sample and tested in a dynamic condition conducted at a heating rate of 10 °C/min, ranging from 20°C to 300°C. After that, the *burn-off cycle* is performed to fully degrade the matrix of each specimen and to determine its resin mass content, following the same procedure as detailed in the Cure kinetic analysis section. The total enthalpy is measured and normalized to the resin mass. Comparing these measurements with the total enthalpy calculated during the cure kinetic analysis, it is possible to determine the residual DoC reached after the isothermal cycle.

Gel point analysis. For the definition of the GP of STR120N131, two different methods are performed to evaluate and compare results. The first methodology is conducted on an Anton Paar MCR 92 rheometer with the same heating rates as DSC analysis from 20°C until the moment in which the samples lost contact with the rheometer plate at around 140°C, performing three repetitions for each condition. The specimens are prepared in a cylindrical shape, each with a radius of 25 mm and 2 mm thick, and are tested at a constant shear rate using a rotational frequency of 1 Hz. Heating is generated from the bottom plate of the instrument and is applied to the lower surface of the sample. Complex viscosity η, storage modulus G', and loss modulus G'' during tests are recorded with 300 sampling points. The crossover point of G' and G'' during thermoset curing defines the GP. This point denotes a transition in the rheological properties of the material, indicating when the elastic behavior becomes dominant over the viscous behavior [17].

The second methodology for defining the GP included employing a high-resolution camera to observe the material flow of a sample situated between two heated plates. Fig. 1 presents a schematic representation of the test setup. The plates and the sample were square in shape, with dimensions of 100 mm and 40 mm, respectively. The specimen consisted of two layers of SMC stacked together, resulting in an overall thickness of around 4 mm. A PTFE film was used to cover the top and bottom surfaces of the material to prevent adhesion between the plates and the sample. Furthermore, it also covered the frontal surface to avoid the flow in the camera direction, thereby maintaining focus on the designated area.

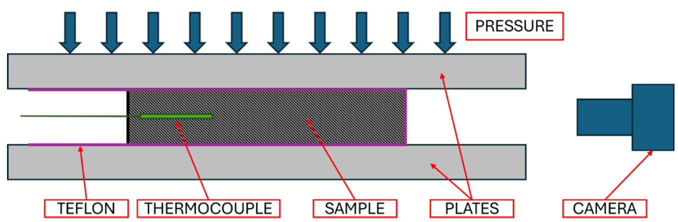

Figure 1 – Test setup for the definition of Gel Point with a high-resolution camera.

The specimens were monitored using a 20-megapixel Blackfly S BFS-PGE-200S6M camera (5472x3648, 8-bit, black-and-white) with a sampling time of 15s. The plates were heated with the same ramp as DSC analysis from room temperature to 180°C. The pressure applied to the specimen during the test is determined by the mass of the plates, which is approximately 1 kg. The gel time was identified visually as the point at which the material ceases to exhibit any detectable flow. A thermocouple was positioned between the two layers of material to monitor the sample temperature accurately throughout the test. In both methodologies, the outcomes obtained from the Kamal-Sourour analysis were utilized to model the evolution of the DoC from the temperature data recorded during the rheological tests.

Result and discussion

Kamal-Souror model. Fig. 2A illustrates the evolutions in time of the DoC. The continuous lines represent the average curves derived from the three repetitions conducted for each heating rate. The dotted lines illustrate the outcomes obtained by fitting the parameters of the Kamal-Sourour model. Each curve has been temporally shifted to align with the start of the cure. It is worth mentioning that higher heating rates lead to a reduced time required to achieve a complete cure for the material. Indeed, considering Fig. 2B, which illustrates the evolution of the reaction rate with the DoC, it can be argued that an increase in heating produces higher velocity in the reaction. The continuous and dotted lines are associated with the same quantities as the previous graph.

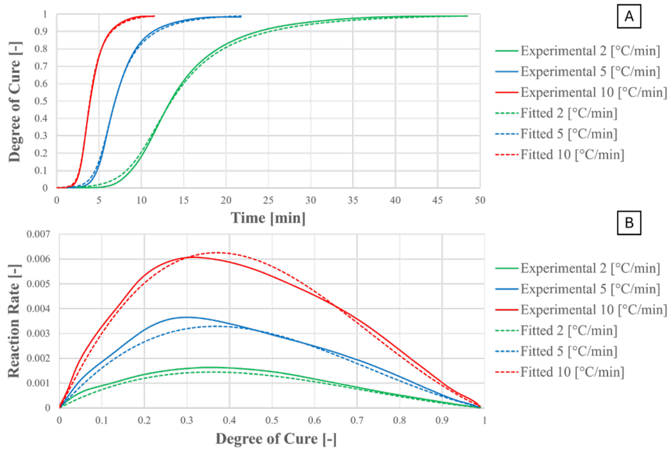

Figure 2 – Cure kinetic results: a) Degree of Cure vs time b) Reaction Rate vs Degree of cure.

By comparing the results, it can be argued that the fitted results exhibit a small overshoot at a heating rate of 10 °C/min while showing a slight undershoot in the other scenarios. A robust correlation is achieved, with an average R^2 value of 99.48% and a standard deviation of 0.05%, determined using the OLS criterion defined in Eq. 4. The coefficients of Eq. 2 and Eq. 3 determined by the best fit for the measured quantities, are shown in Table 1.

Table 1 – Coefficients of the Kamal-Sourour model of Eq. 2 for STR120N131 prepreg

Constant	Value	Units
A_1	2.212E+05	[s^{-1}]
A_2	8.068E+13	[s^{-1}]
E_1	1.549E+05	[J/mol]
E_2	1.240E+05	[J/mol]
m	6.410E-01	[-]
n	2.613E+00	[-]

The results of the validation analysis of the Kamal-Sourour model are listed in Table 2. The predicted values are derived from the parameters presented above, considering the final DoC values reached at the conclusion of the isothermal cycle. The average measurements and corresponding standard deviations of the effective residual DoC values, obtained from the three repetitions of dynamic DSC scans, are presented in the third column.

Table 2 – Results of the validation of the fitted parameters for the Kamal-Sourour model.

Cycle temperature	Predicted residual DoC	Measured residual DoC
100 [°C]	49.45 [%]	51.79 ± 3.08 [%]
120 [°C]	11.07 [%]	6.27 ± 0.75 [%]
140 [°C]	3.64 [%]	0.0 [%]

The isothermal cycle conducted at 140°C exhibits, in each repetition, no residual enthalpy, indicating that the sample is completely cured. The average mismatch between the two values is around 3.6%, demonstrating a robust validity of the predicted data obtained using the Kamal-Sourour model and the identified parameters.

Gel point analysis by rheometer test. Fig. 3 illustrates the evolution of complex viscosity, dynamic moduli, and DoC in relation to the temperature.

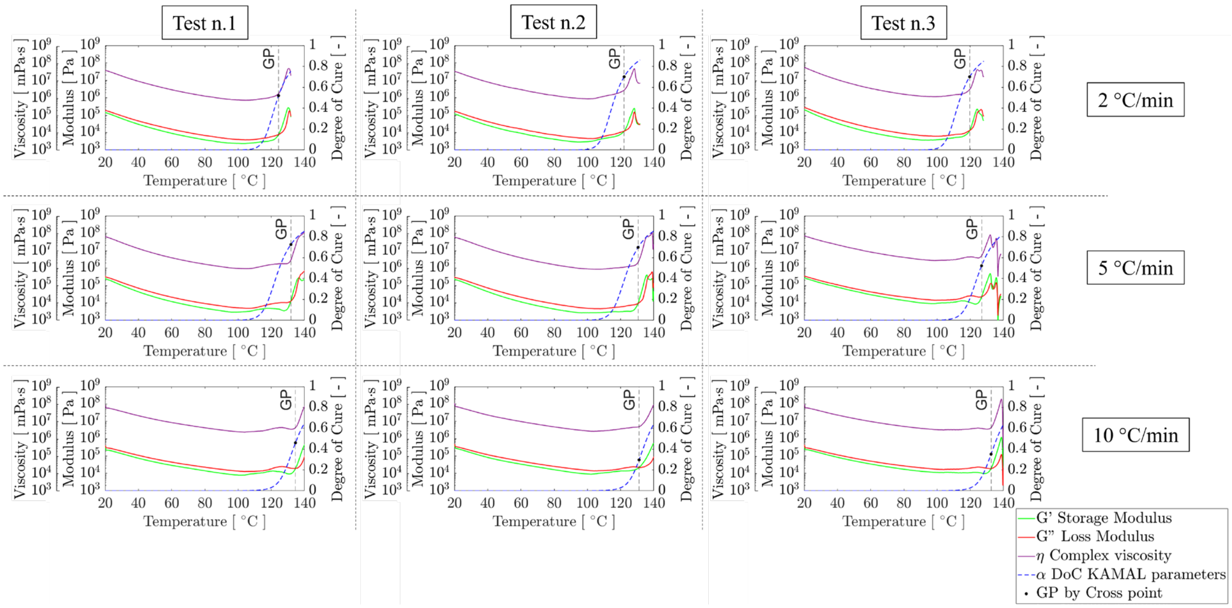

Figure 3 – The evolution of Complex viscosity, Storage Modulus, Loss Modulus, and Degree of Cure during rotational rheometer testing.

The columns differentiate between the experimental runs performed, while the rows represent the different heating rates applied during the tests. In the initial stages of each experiment, the material is in a liquid state, where viscous behavior predominates. This is followed by:

$$G'' > G' \tag{5}$$

The two moduli and viscosity decrease as the temperature rises until a minimum, leading to increased mobility of the resin molecules. Reaching a temperature around 100 °C, the prepreg starts the curing reaction, and these quantities rise. As the reaction progresses, the moduli increase with different velocities, corresponding to the increasing cross-link density and molecular weight of the curing polymer system. At around a temperature of 120-130°C, the storage modulus crosses the loss modulus, defining the gel point, identified by the black point in each graph. Beyond this point, the SMC changes phase, shifting from liquid to rubbery. It can be argued that the moduli and viscosity increase exponentially as the reaction advances. Following this stage, it is worth noting a subsequent reduction in moduli and viscosity. This phenomenon can be attributed to the loss of contact at the interface between the material and the rotating plate. Lastly, it is worth mentioning that the viscosity at the gelation does not exhibit an abrupt increase in values. Indeed, it may be possible to define the GP as the moment in which viscosity approaches practically infinite values. However, as reported by E. Hanna et al. [16], this second methodology exhibits reduced sensitivity to variations in temperature, leading to an overestimation of measured gelation time values.

The specific value α_{gel}, of DoC at which the GP is reached, calculating by crossover point, can be identified in each test. It is important to note that no clear correlation can be established between α_{gel} and the heating rate. The average value and the standard deviation calculated in the entire campaign are:

$$\alpha_{gel\_RHEOM} = 55.32\% \pm 16.20\,\% \tag{6}$$

Gel point analysis by camera analysis. Fig. 4 presents the temperature evolution recorded by the thermocouple, along with the DoC calculated using the Kamal-Sourour model based on this data set.

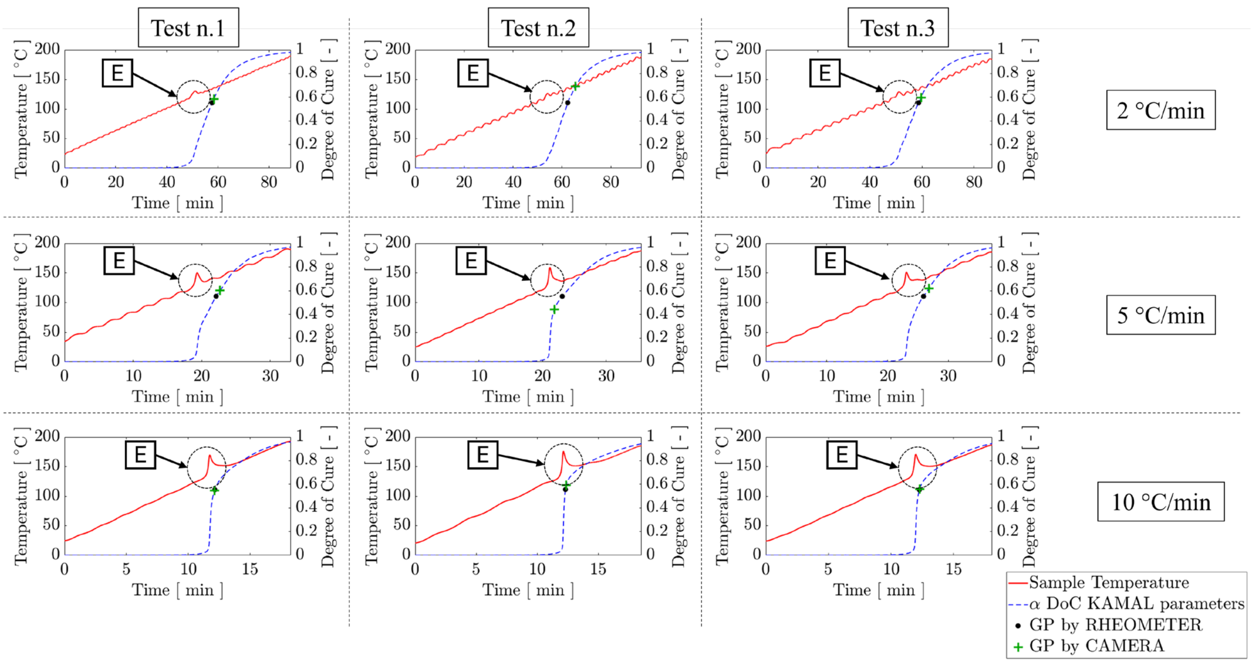

Figure 4 – Evolution of temperature and Degree of Cure during the Gel Point tests with a high-resolution camera.

Examining the sample temperature, as highlighted by the dotted circle and identified with E, it is possible to observe a peak that deviates from the temperature trend. This exhibits a maximum value correlated with the heating rate. Specifically, higher velocities result in greater peak amplitudes. It can be argued that this phenomenon can be attributed to heat release during the exothermic reaction associated with the curing process. Indeed, a faster reaction produces more released heat flow and, consequently, higher maximum temperature values monitored at the peak [18]. Considering the evolution of DoC, represented by the blue dotted line, it is possible to mark the α_{gel} based on the data obtained from rheometer tests, identified by the black point in each plot. The corresponding quantity can be determined by analyzing images captured by the camera, represented by the green mark. Also, in this case, it can be argued that no distinct correlation has been observed with the heating rate. It is worth mentioning that the results from the two methodologies consistently demonstrate a high level of agreement.

Fig. 5 presents a series of frames from test n. 1, conducted at a heating rate of 10 °C/min. These images illustrate the flow evolution as recorded by the camera near the GP. Fig. 5A-B illustrate the flow of material before reaching the GP, Fig. 5C displays the moment at which the GP is reached, while Fig. 5D represents the subsequent frame captured by the camera.

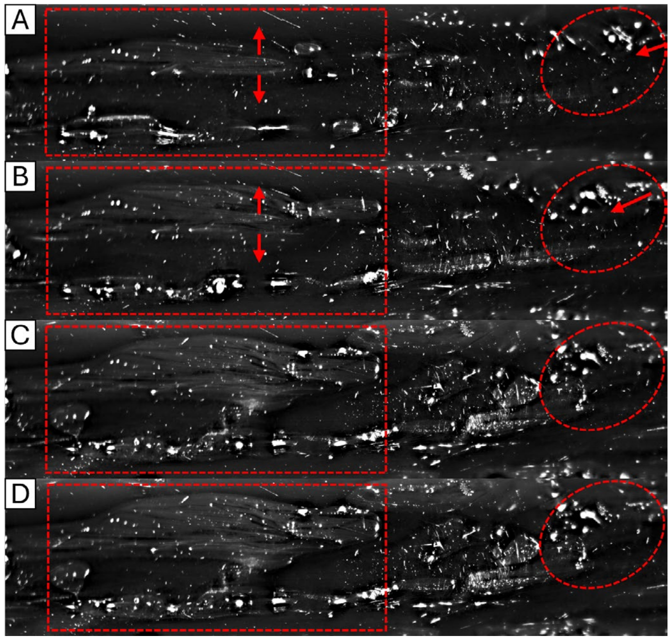

Figure 5 – Test N. 1 conducted at a heating rate of 10°C/min: A) Two frames before GP; B) One frame before GP; C) Frame at GP; D) One frame after GP.

The areas highlighted by the dotted red box and circle illustrate the observable flow of the material. In Fig. 5A, the two arrows within the rectangle illustrate the progression of the material movement throughout the subsequent steps. Indeed, it is worth mentioning that the grey area within the box enlarges in size as it progresses from the first to the third step. Following the step depicted in Fig. 5C, the GP has been reached, and no detectable flow can be observed in the next photo. Considering the dotted circle, the arrow indicates the movement of several bubbles within the highlighted area. It can be argued that these bubbles progressively shift to the left, ceasing their movement upon reaching the GP in the third frame. Indeed, comparing Fig. 5C-D, no differences can be detected. Considering all tests performed, it is possible to calculate the average value and the standard deviation of α_{gel} by camera analysis:

$$\alpha_{gel\_CAMERA} = 58.20\% \pm 6.71\% \tag{7}$$

Conclusion

The capability to accurately model the Degree of Cure can significantly impact molding time, as it helps prevent materials from remaining in the mold for longer than necessary, significantly reducing production costs. The parameters of the Kamal-Sourour model estimated in this work through the Ordinary Least Squares method enabled an accurate prediction of the evolution of the Degree of Cure. The validation of the fitted parameters shows a strong agreement between the predicted and measured values of the Degree of Cure, with a final error lower than 4%. Based on these data, this study has successfully demonstrated a viable alternative technique for identifying the Gel Point of carbon fiber SMC materials through a comparative analysis with conventional rheometer methods. The Gel Point is directly associated with the flowability of the material, its capacity to fill the mold, and the potential formation of defects. The capability to monitor it through the crossover point of the Storage and Loss modulus in oscillatory analysis has been largely demonstrated in the literature. Therefore, the results are compared with an alternative methodology that employs a high-resolution camera capable of capturing the flow of the material positioned between two heated plates and recording the Gel Point. The outcome comparison reveals a strong agreement between the two cases, with a mismatch of approximately 3% in α_{gel}. Furthermore, the analysis performed using the rheometer test setup demonstrates a higher variability in the final value of GP compared to the high-resolution camera method. This variability may be attributed to the small size of the specimen, which has a diameter of 25 mm, or to the inability to achieve uniform heating across both the top and bottom surfaces of the specimen. The proposed novel method offers a clear and effective approach for defining the Gel Point of these materials, guaranteeing consistent outcomes characterized by a smaller variability when compared to traditional analysis techniques commonly utilized for defining this quantity.

Founding
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