SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage: https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879

A Rheological Study of Fibre Reinforced Composites and The Factors That Affect Rheological Behaviour During Impregnation Process: A Review

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ARTICLE INFO

Article history:
Received 29 June 2021
Received in revised form 30 October 2021
Accepted 3 November 2021
Available online 4 December 2021

Keywords:
Rheological; pre-impregnation; fabric; fibre reinforced composites

ABSTRACT

Rheological behaviour is an important factor affecting the flow behaviour of a fluid and many aspects related to this, mainly in the manufacturing process of fiber reinforced composites, either for Newtonian fluids or non-Newtonian fluids. During impregnation process, the viscosity changes with temperatures and their strain rate, has influenced the resin flow behaviour during curing process. In this paper, a review on the rheological studies of fiber reinforced composites for both, synthetic and natural based fibers, respectively, are presented. In addition to that, this review paper highlighting a few research studies conducted in literature on the main factors that affecting the rheological quality and performance of the composites. The aims of this review, mainly to capture the trend ranging from the recent five years back and summarize the various studies via experimental, theoretical or modelling works. Furthermore, also aiming to provide an ideal baseline information in the selection of the methods regarding rheological study to ensure better quality of pre-preg product and fibre reinforced composites can be produced in the author's future work.

1. Introduction

Composite materials have been widely used in the engineering industries such as automotive, aerospace, and marine industries. Such industries favour the composite material for its lightweight properties, high durability, and corrosion resistance, yet still offers a strength as similar as other materials such as steel. These factors make composite material economically friendly and gives a longer lifespan. However, some composite products have difficulties in manufacturing as the fibre/resin is required to be reinforced during manufacturing in achieving fully consolidated state [1].

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https://doi.org/10.37934/arfmts.89.2.167181

A rheological study is defined as a study of flow behaviour of liquids and the deformation behaviour of solids, in which the deformation produced by shear forces cause the materials to flow. The rheological behaviour may be influenced by various factors such as; type, degree and duration of loading in terms of velocity or force applied, or shear rate and shear stress and temperature [2]. The study of viscoelastic fluid behaviour during various manufacturing process have been discussed as an important subject matter by various researchers in understanding the fluid flow and heat transfer problem, mainly for non-Newtonian fluid [3].

In the scope of this study, the rheological behaviour of the resin during the impregnation of pre-impregnated (pre-preg) and heat transfer during manufacturing of the fibre reinforced composites using Out of Autoclave (OoA). With regard to the establishment of the pre-preg laminating method, which refers to process of impregnating the fabric reinforcement with the resin in manufacturing the pre-impregnated (pre-preg) and their composites, further study considering the resin flow through the fabric weave tightness and crimpness for example, need to be addressed. There are few factors and limitations contributing to the quality and performance of the product produced. Therefore, the impregnation quality of natural based reinforced pre-impregnated (pre-preg) would need to be further investigated with regard to the rheological behaviour of the resin, mainly in reducing the cost of the experimental works and consumable materials. It is aimed from this review, providing some baseline to the selection of the most suitable methods either for theoretical and/or modelling and experimental methods. The outcomes from this review would possibly provide some insight of choosing the appropriate rheological methods specifically, to be established with the respective thermoset based resin materials and their fabric reinforcement in the author's future works.

2. Rheology Study

Rheology properties such as initial viscosity, minimum viscosity, and gel point are important to determine the behaviour of a resin. initial viscosity indicates the onset of matrix melting and is not influenced by heating rate. It is a useful indicator of degree of resin advancement for quality control. Minimum viscosity describes the period and temperature range over which resistance to flow is lowest. In the laminate production, the resin viscosity must be low enough to wet the embedded fibres uniformly, but not so low as to cause excessive bleeding at the laminate edges. There are two methods in studying the rheological properties of a resin which are, via theoretical prediction and modelling work and via experimental method, are reviewed here.

2.1 Theoretical Prediction and Rheological Modelling Works

In the past few years, rheological modelling has made it possible for these structures to be more scientifically analysed which has contributed to a decrease in design and raw material costs. A summary of various research papers on rheological studies via modelling and/or theoretical prediction works is tabulated in Table 1. Rheological modelling is used to predict the start parameters and selected regression algorithm. The model is usually related to the gelation of a resin. These models are often constructed to compare it with experimental data conducted. From these models, a gel point criterion can be observed through the data results. Various researchers have created empirical modelling to characterize these criteria according to the situation of the test. The model can be divided into two classifications, temperature dependant and time- dependant. These models are based on various mathematical equation presented to predict the parameters included and selected regression algorithm. The models used are then compared to the results from experimental results conducted. Through comparison, the characteristics of a resin can be seen and concluded before the experimental work being carried out.

Table 1
Recent research papers on rheological studies via modelling and/or theoretical prediction

Rheological Model	Researchers and Model
Temperature-dependant	Maji *et al.,* [4] (Broyer and Macosko equation)
	Xue *et al.,* [5] (Arrhenius relationship)
	Y. Chen *et al.*, [6] Kinetic Model
	Rzek *et al.*, [7] (Arrhenius relationship) (William–Landel–Ferry equation)
	Perego *et al.*, [8] (William–Landel–Ferry equation)
	Puentes *et al.*, [9] (Castro-Macosko model)
	Abliz *et al.,* [10] (William–Landel–Ferry equation)
	Apostolidis *et al.,* [11] (Arrhenius equation)
	Geissberger *et al.,* [12] (Castro-Macosko model)
	Garcia-Martinez *et al.,* [13] (William–Landel–Ferry equation)
	Abliz *et al.,* [14] (Castro-Macosko model)
Time-dependant	Romberg *et al.,* [15] Buckling model
	Djebara *et al.,* [16] (Carreau empirical model)
	Asiaban *et al.,* [17] (Bingham Model)
	Ma *et al.,* [18] (Power law model)
	J Zheng *et al.,* [19] (Power Law Model)
	L. A. Shah *et al.,* [20](Bingham model, Modified Bingham Model, Ostwald power law model)
	Suiker [21] Buckling model
	Javidi *et al.,* [22] (Ellis model)
Time and temperature dependant	Rique *et al.,* [23]
	Garschke *et al.,* [24] (Castro-Macosko, Empirical model)
Others	W. Yang *et al.,* [25] Kinetic Model (Kamal Model)
	Cavdir *et al.,* [26] Shear load-dependant (Newtonian Model)
	Kotula *et al.,* [27] Frequency-dependant (GEM model)
	Hauswirth *et al.,* [28] Shear-dependent dynamic viscosity (Cross model)
	Laurien *et al.,* [29] Flory-Stockmayer Equation
	Harkous *et al.,* [30] Kinetic Model (Kamal Model)

2.2 Experimental Works

In experimental method, the samples are subjected to temperature and shear generated from the rheometer oscillatory geometry. This method is used to study the reaction of a resin and how it can affect the characteristics of it. The testing method then provide information regarding initial viscosity, minimum viscosity, Gel point, and optimum heating rate [31]. A summary of various research papers on rheological studies via experimental works using various methods, mainly using rheometer and viscometer, is tabulated in Table 2. Since gel point occurs around the glass transition temperature, thermal measurements are conducted to support the gel point measurement process. This determination of these parameters can be achieved with a help of Differential Calorimetry Scanning (DSC), in which be used by various researchers. The DSC results provides information regarding the initial curing temperature, peak, final curing temperature and glass transition temperature, in which help in the setting and selection of the experimental parameters to be used for the impregnation and manufacturing process.

Table 2
Research papers on rheological studies via experimental works using various viscometer methods

No	Author (Year)	Geometry	Scope of Study and Measurement method
1	W. Yang *et al.,* [25]	Parallel plate	• Temperature ramp rheology test • Isothermal rheology test
2	Hiremath and Kumar Shukla [32]	Parallel plate	• Dynamic strain sweep with various shear rate. • Frequency sweep tests in stress-controlled mode.
3	Dorr *et al.,* [33]	Parallel plate	• Time sweep test • Multiwave test
4	Fang *et al.,* [34]	Parallel plate	• Small-amplitude oscillatory shear test • Multifrequency
5	Liang *et al.,* [35]	Parallel Plate and Cone-Plate	• Dynamic strain sweep test • Nonlinear rheological measurements
6	Nobile *et al.,* [36]	Parallel plate	• Strain sweep test • Frequency sweep tests
7	O Dagdag *et al.,* [37]	Parallel plate	• Frequency sweep test at isothermal condition
8	O Dagdag *et al.,* [38]	Cone-plate	• Shear viscosity test
9	Mai *et al.,* [39]	Parallel plate	• Oscillatory shear test • Frequency sweep test
10	Mphahlele *et al.,*[40]	Parallel plate	• Oscillatory shear flow measurements under isothermal and dynamic conditions • Preliminary frequency and strain sweep tests.
11	Patel *et al.,* [41]	Parallel plate	• Frequency and flow sweep tests.
12	Y. Zhang *et al.,* [42]	Parallel plate	• Time sweep test under the oscillatory condition
13	Martinez-Miranda *et al.,* [43]	Parallel plate	• Oscillatory test at isothermal and non-isothermal condition • Constant gap (plate-plate separation) and constant normal force • Multifrequency test • Multiwave test
14	McKenzie *et al.,* [44]	Parallel plate	• Strain amplitude oscillatory shear sweeps test at non-isothermal condition
15	Stroble *et al.,* [45]	Parallel plate	• Strain sweep test at isothermal condition
16	Rezvani *et al.,* [46]	Cone-plate	• Strain amplitude sweep test • Small Amplitude Oscillatory shear (SAOS) frequency sweep tests
17	O Dagdag *et al.,* [47]	Cone-plate	• Shear viscosity test in non-isothermal condition
18	Rafiee *et al.,* [48]	Parallel plate	• Steady shear test
19	Liu *et al.,* [49]	Parallel plate	• Strain sweep test
20	Vijayan P *et al.,* [50]	Parallel plate	• Dynamic frequency sweep test
21	Abliz *et al.,* [14]	Parallel plate	• Dynamical viscosity measurement • Isothermal viscosity measurement • Oscillation viscosity measurement
22	Garcua-Martinez *et al.,* [13]	Parallel plate	• Strain sweep tests at different constant temperatures and frequencies. • Multifrequency test in isothermal condition.
23	Chaloupka *et al.,* [51]	Parallel plate	• Constant deformation at an angular frequency • Frequency sweep tests
24	Compton *et al.,* [52]	Parallel plate	• Oscillatory stress sweeps test • Flow sweep test • Constant-shear conditioning step
25	Adrar *et al.,* [53]	Parallel plate	• Strain sweep test
26	Nasab *et al.,* [54]	Parallel plate	• Strain sweep test • Frequency sweep tests

27	L. Wang *et al.,* [55]	Concentric cylinder and Parallel Plate	• Steady-state test at shear rate of 0.1 to 100 s-1 • Oscillatory frequencies range from 0.1 to 100 hz • Strain sweep test

While, rheological test provides some information with regards to the shear flow rate and viscosity at various iteration of temperature, pressure and frequencies. The type of geometry used also being summarised accordingly, as can be seen in Figure 1. The use of parallel plate is widely applied for the measurement using viscometers, mainly for the works with the thermoset resin, especially when the rheological study during curing conditions, the parallel plate showed the most suitable geometry.

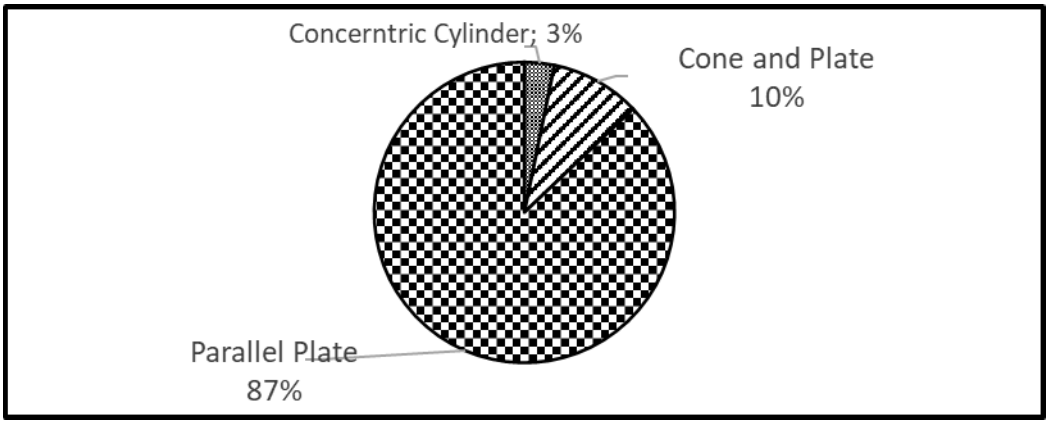

Fig. 1. Research papers studied process using various types of geometry of viscometer [13, 14, 25, 32-55]

3. Factors that Affect the Rheological Behaviour

3.1 The Study of Impregnation Process Using Various Types of Resin and Fibres

As the composite manufacturing and development is vastly improved, fiber reinforcement such as in fabric form can be resin-impregnated before manufacturing in a separate process. This method is called pre-impregnation process. A pre-impregnated composite can be manufactured in many different techniques. The process produces pre-preg as a semi-finished product, where the fabric reinforcements pre-impregnated with matrix, producing semi-finished materials that are ready to be applied in production line. Nowadays, pre-pregs are becoming widely used in the industries due to time saving and easy application during any composite related repair procedure. With the blooming usage of pre-preg materials, many companies had started manufacturing and supplying these materials to the user. Relative to that, this research will be introducing natural-based fabric as an alternative reinforcement material for pre-pregs manufacturing. A few research studies conducted investigation with regard to various manufacturing process which involving impregnation stage is summarized and tabulated in Figure 2.

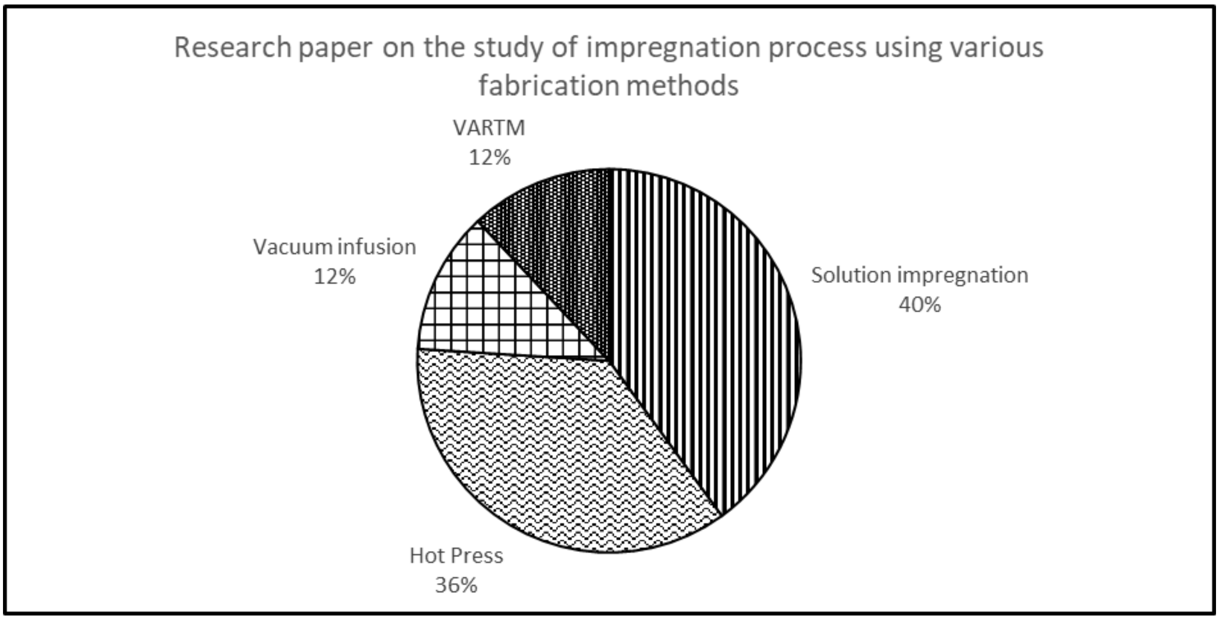

Fig. 2. Research papers related to the study of impregnation process in fabrication of fibre reinforced composites using various methods [73, 83-98]

Pre-pregs usually consists of two main elements, which are the matrices and fibre reinforcements. The combination of these two elements will give pre-pregs unique physical and characteristic. Polymeric nature resins are commonly used as pre-preg matrices because it can be synthesized with multiple chemicals according to required characteristic. Resin is responsible for combining reinforcements to provide interlaminar shear strength. Resin is sensitive toward environmental changes such as temperature, moisture, and light radiation that could change the properties especially after being fabricated with reinforcements. In order to get a proper characterization of resin, it is crucial to have better understanding on the behaviour of the resin when exposed to the environment. Another main factor is reinforcement materials, which are the main component of pre-pregs that providing the strength of pre-pregs. These materials usually exist in fibres and fabrics form depending on the usage and application in the industries.

A pre-preg composite can be found in two stages: Semi-cured product where the pre-pregs are stored in rolls into the freezer, and in fully cured product in application of prepreg in mould. These two stages can be related to the curing stage of a resin. The resin in pre-preg process can be classified into two types, thermoset, and thermoplastic resin. The thermoset is much favourable resin to be used in pre-preg as it offers a desirable tack and drape to allow more complicated shapes to be formed rather than just flat plates [56]. Furthermore, thermoset offers a high thermal and mechanical properties compared to thermoplastic. Many types of thermosets can be found in the manufacturing of prepreg. Figure 3 and Table 3 show a summary of few research papers conducted their study with regards to the behaviour using various types of thermosets based-resin. It can be seen that; the most common and popular choice of resin is focussed on epoxy-based resin due to the several advantages offered by epoxy resin. In several industries, epoxy resin systems are commonly introduced, from adhesives, coatings, to high-performance matrix systems fiber-strengthened composites. Like the other thermoset resins, epoxy final properties depend on its cure process and the final cure state. Thus, the inability to accurately design the curing process may lead to a poor fibre/matrix bonding, large residual stress, and can even lead to thermal degradation of the matrix due to high temperature [57].

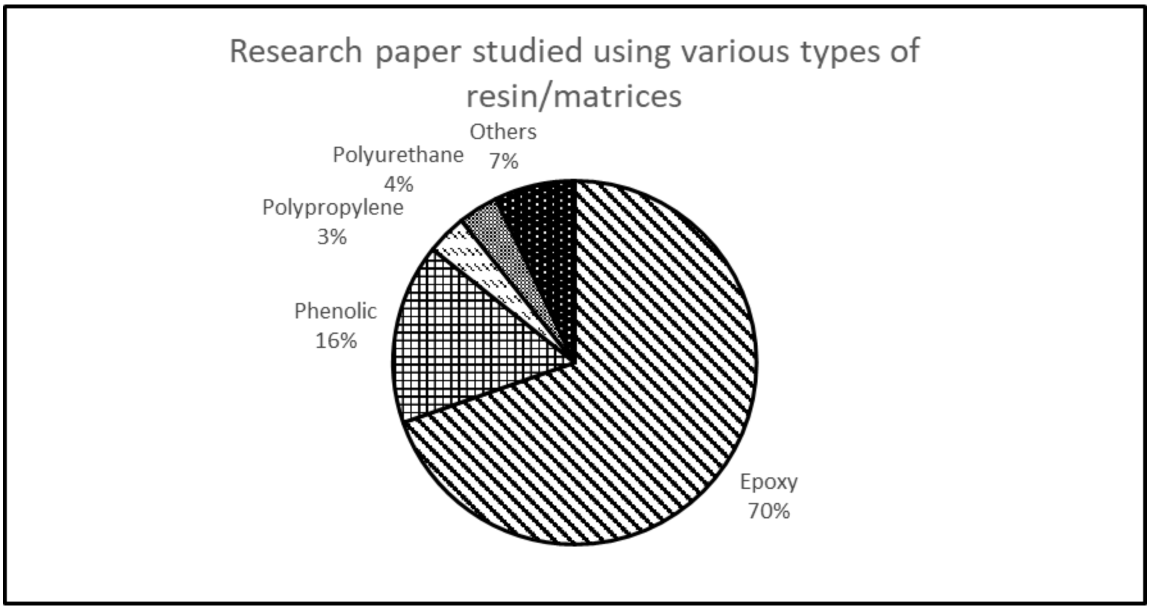

Fig. 3. Research papers studied using various types of resin or matrices

Table 3
Research papers on rheological studies using various types of thermoset

No	Author	Type of Resin
1	Feghali *et al.,* [58]	Biobased Epoxy resin
2	Pouladvand *et al.,* [59]	DGEBA Epoxy and EPON828 Epoxy resin
3	H. Li *et al.,* [60]	Epoxy Resin
4	Gbadeyan *et al.,* [61]	LR 30 and LH 30 Epoxy resin
5	Memon *et al.,* [62]	AFG-90H Epoxy resin
6	Walte *et al.,* [63]	HSC 7660 Epoxy resin
7	Shi *et al.,* [64]	Epoxy Resin
8	Raju *et al.,* [65]	LY556 Epoxy resin
9	C. Zheng *et al.,* [66]	Phenolic Resin
10	S. Yang *et al.,* [67]	DGEBA Epoxy resin
11	S. Y. Kim *et al.,* [68]	EPON828 Epoxy resin
12	Austermann *et al.,* [69]	Epoxy-based photopolymer resin EPX81
13	K. Zhang *et al.,* [70]	DGEBA E51 Epoxy resin
14	Shukla *et al.,* [71]	LY556 Epoxy resins
15	Jenkins *et al.,* [72]	IN2 Epoxy
16	Ekrem *et al.,* [73]	DGEBA Epoxy resin
17	Zhao *et al.,* [74]	DGEBA Epoxy resin
18	Lei *et al.,* [75]	Modified resol phenolic resin
19	Billisik and Sapanci [76]	Araldite EPN 1138 Phenolic Resin
20	Ciesielski *et al.,* [77]	DGEBA Epoxy resin
21	Asaro *et al.,* [78]	Ammonium-modified phenolic resin
22	Martins *et al.,* [79]	DGEBA Epoxy resin
23	M. Li *et al.,* [80]	Epoxy resin E51

3.2 Main Factors that Affecting Rheology Behaviour

As the matrix goes to a curing state, several properties may change due to the chemical reaction acting to it. One of the important parameters that highly impacts the flow behaviour of a resin is viscosity [2]. The viscosity of a resin changes due to the factors affecting them such as time, temperature, and shear rate. This describes the fluid flow of a resin. however, viscosity of a resin is not enough in describing the behaviour of a resin as it goes through gel point, making it a viscoelastic material. Therefore, rheology properties must be studied to determine the flow of a resin from liquid to solid state. In the process of manufacturing a prepreg composite, the most critical process is resin flow penetration in the fibre web. The factors affecting the resin impregnation rate can be analysed in Darcy's Law [81].

$$V = \frac{k_f}{\mu(T)} * \frac{dP}{dY} \quad (1)$$

Darcy's Law is an equation that describes the flow of a fluid through a porous medium. In impregnation process, Y describes impregnation rate in thickness, V describes the flow velocity, μ describes the resin viscosity, k_f describes the fibre bed permeability, and P describes the applied pressure. This law is much related with the permeability of the fibre composite. However, the rheology properties must be studied in accordance with the law as resin flow is affected by the temperature and pressure of the pre-impregnation process as well [81].

In a composite pre-impregnation process, a resin/fibre goes through curing stages from a liquid to a solid state affected by certain factors such as temperature, shear rate, pressure, and time. These factors are studied to form rheological model to predict the behaviour of a resin. As stated above, Darcy's Law is commonly used to describe the resin flow in pre-impregnated process. Other relationships are needed to describe the permeability and viscosity of resin. Another concept that has been developed is Pre-preg Flow Number (PFN) [82].

$$\mathrm{PFN} = \frac{\mathrm{KP_{eff}}}{\mathrm{\mu V Y}_f} \quad (2)$$

PFN is a concept that describes the interrelationship of temperature, pressure, and production rate of a prepreg [82]. These are the critical parameters that can affect the quality of a pre-preg. Other empirical models can be found to study the rheological behaviour of a resin as well. These models are categorised into two factors, temperature, and shear rate. The viscosity of a resin changes according to the factors reacted to it. Depending on the characteristics of a resin, the temperature changes the viscosity until it reaches a point where it becomes glassy state, this is called the glass transition region. The glass transition can further identify one of the rheology characteristics which is gel point. Other characteristics that can be described during this stage is initial viscosity and minimum viscosity. Beyond that, viscosity behaviour cannot be described as the resin changes its state to a viscoelastic state.

With regard to the determination of viscoelastic properties and their effects on the resin properties, the storage modulus, G', loss Modulus, G'' are the main parameters that describes the rheological behaviour of a thermoset. The flow curves determine the viscoelastic behaviour of a resin, as thermoset goes through three curing stages that are irreversible, it is important to analyse the viscoelastic behaviour of a thermoset as well as the factors acted upon it.

In the rheology of thermoset based-resin, the gel point is important in determining the time and temperature at which pressure should be applied. Gel point is a temperature or time where the first group of covalent bonds binds within the sample and becomes infinitely large in molecular weight [31]. The gel point of a thermoset occurs during curing process, near the glass transition temperature process. The common criteria shown to identify the gel point is the crossover of G' and G'' [42]. However, it is limited to a characteristic subjected to frequency dependant event, and only if the system is stoichiometrically balanced and the curing temperature significantly above its Tg. There are few other researches, which investigated various ways of identifying gel point of a thermoset during curing process. Another criterion that is acceptable in identifying gel point when the temperature dependence of tan δ becomes frequency independent [43]. As for the summary, Figure 4 show the rheological studies using various criterions to determine gel point and viscoelastic properties.

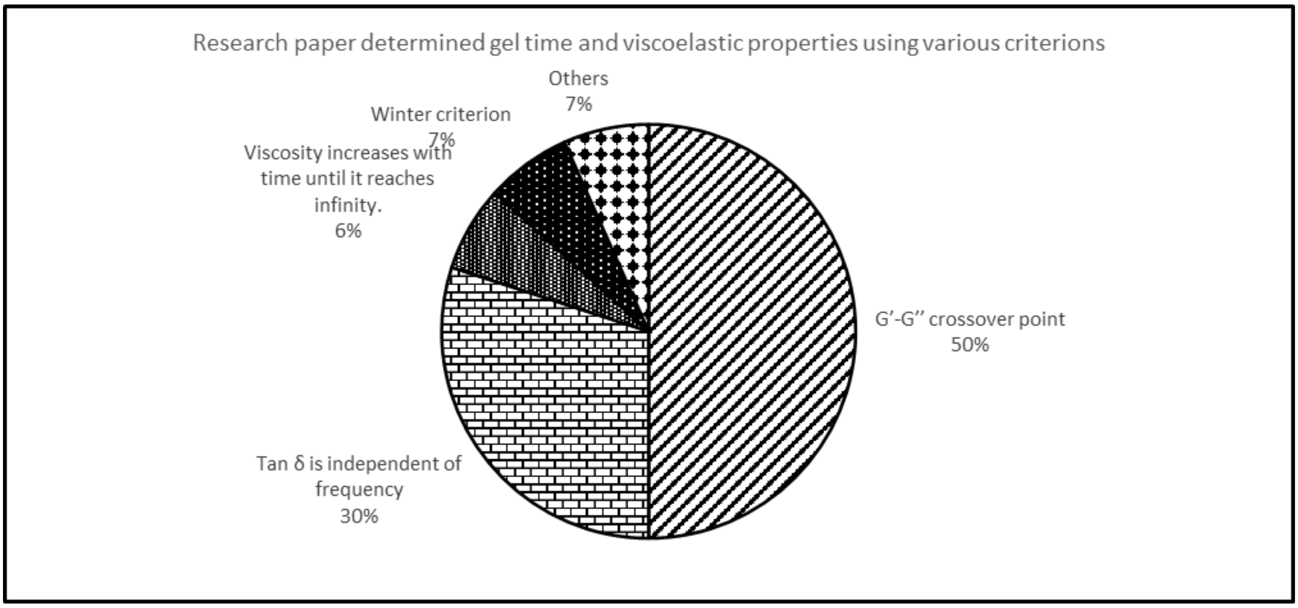

Fig. 4. Research papers on rheological studies of thermoset based-resin using various criterions focusing in the determination of gel point and viscoelastic properties [5, 9, 13, 14, 25, 30, 32, 33, 40, 42-45, 99-108]

4. Conclusion

It can be concluded that the selection of the most suitable rheological model plays a big role in estimating the start parameter in order to carry out the next stage for the experimental part. The measurement methods and the factors affecting the rheological behaviour of a thermoset have been summarized from the previous papers. Various experimental and theoretical and/or modelling works has been summarized and reviewed as tabulated, with regard to various manufacturing process which involving impregnation stage. The rheological studies of thermoset based-resin using various criterions in determination of gel point and viscoelastic properties have been reviewed in order to understand the time of occurrence of gel point can accurately determine the curing process and final cure state, therefore improving the product quality of fibre/matrix and this could enhance the selection of methods stage and providing a baseline study for the research works related to this scope.

Acknowledgement
This research was funded by an internal grant, (STR 19029), Short Term Research Grant (STRG) from Universiti Kuala Lumpur.

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