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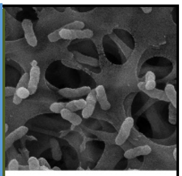

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Outlier and Normality Testing of the Residuals from the Carreau-Yasuda Model in Fitting the Rheological Behavior of the Non-Newtonian fluid TF2N

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ABSTRACT

Non-Newtonian fluids include a variety of regularly encountered substances such as custard, honey, toothpaste, starch suspensions (including starch from corn starch), paint, blood, melted butter, and hairspray. For decades, scientists have investigated non-Newtonian fluid behavior and produced models to aid in the characterization of non-Newtonian fluid behavior. In addition to data interpolation and extrapolation, the outputs of these models may be utilized for material classification based on model parameters and aid with the simulation of computational fluid dynamics. The Carreau-Yasuda model fitted to the rheological behavior of the non-Newtonian fluid1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide ([emim][TF2N]) was checked for its conformation to the normal distribution of its residual using the normality tests , which was found not to pass all of the test. After checking for the presence of an outlier using the Grubbs ‘test, no outlier was detected. The ROUT method was then applied to detect the presence of outliers and three outliers were found and removed. The normality checks performed on the cleaned residues gave acceptable results in terms of normality tests and visual conformation of the residual, Q-Q plot and overlaid normality curve to the histogram, indicating that the model is now appropriate for the data.

INTRODUCTION

The ability to make decisions based on current data analysis is critical throughout the whole research and development process. Many fluids on are non-Newtonian and, as a result, rely on their viscosity to function properly. The method by which non-Newtonian rheological data is measured, corrected, and analyzed is critical in the process of formulating new materials and processes [1–4]. When a fluid does not comply with Newton's law of viscosity, it is said to be non-Newtonian. The law of viscosity says that viscosity should remain constant regardless of stress. It is possible for non-Newtonian fluids to transition from a liquid to a solid state when they are pushed. During the shaking process, ketchup, for example, becomes runnier and therefore becomes a non-Newtonian liquid [3,5–9]. When dealing with non-Newtonian fluids, the viscosity (progressive deformation produced by shear or tensile stresses) is frequently impacted by the shear rate or by the history of shear rate. Certain non-Newtonian fluids with shear-independent viscosity, on the other hand, exhibit normal stress differences or other non-Newtonian properties, despite their shear-independent viscosity. It is linear, passing through the origin, between shear stress and shear rate in a Newtonian fluid with the constant of proportionality being the coefficient of viscosity as the constant of proportionality. In a non-Newtonian fluid, the relationship between shear stress and shear rate is not the same as in a Newtonian fluid. It is possible that the viscosity of the fluid will vary over time. The conclusion is that establishing a consistent coefficient of viscosity is a near-impossible task [10–14].

In non-Newtonian and Newtonian fluids, hydrogen bond plays a major role in their behavior [15]. As ana example, the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]) shows a Newtonian plateau followed by a second shear thinning where the reduction in viscosity is observed. This behaviour is dependent upon hydrogen bonding where the reduction in viscosity (as modelled using the Carreau-Yasuda) is proportionately to a decrease in the number of hydrogen bonds [16]. Like all curve fitting exercise, a nonlinear regression like the Carreau-Yasuda model using the least square method must obey one of the rules of nonlinear regression, where the residuals of the curve must be normally distributed, and the residuals must be checked for the presence of outliers [at 95 or 99 percent confidence levels]. In most cases, normality tests such as the Kolmogorov-Smirnov, Wilks-Shapiro, Anderson Darling and D'Agostino-Pearson, as well as the Grubb's test, which tests for the presence of an outlier, are usually used, and these are the objectives of this study.

METHOD

Data for the Rheological flow curve of the ionic liquid as modelled using the Carreau-Yasuda model (**Figure 6**) from a published result [16] was extracted using the WebPlotDigitizer 2.5 software [17] which helps to digitize scanned plots into table of data with good enough precision [18].

Residuals

Residuals are very important in assessing the health of a curve from a particular used model. Mathematically, residual for the i^{th} observation in a given data set can be defined as follows (Eqn. **1**);

$$e_i = y_i - f\left(x_i; \hat{\beta}\right) \qquad \textbf{(1)}$$

where y_i denotes the i^{th} response from a given data set while x_i is the vector of explanatory variables to each set at the i^{th} observation corresponding values in the data set.

Grubbs' Statistic

In an average value, a single data point with deformation can lead to gross error in the fitting of a nonlinear curve. Therefore, searching for an outlier is an integral aspect of curve fitting. The Grubbs test is used to evaluate the outlier in the univariate environment and the data is normally distributed [19]. The test can be applied to the maximal or minimal observed data from a Student's t distribution (**Equation 1**) and to test for both data simultaneously (**Equation 2**). In this test, reject the point as an outlier if the test statistic is greater than the critical value.

$$G_{\min} = \frac{\bar{X} - \min(X)}{s}$$

$$G_{\max} = \frac{\max(X) - \bar{X}}{s} \qquad \textbf{(1)}$$

$$p_G = 2n.p_t\left(G\frac{\sqrt{n(n-2)}}{n-1}, n-2, 1\right)$$

$$G_{\text{all}} = \frac{\max(\bar{X} - \min(X), \max(X) - \bar{X}}{s}$$

$$p_G = n.p_t\left(G\frac{\sqrt{n(n-2)}}{n-1}, n-2, 2\right) \qquad \textbf{(2)}$$

In the event the Grubbs test failed to detect an outlier and the residuals are shown to be overwhelmingly nonnormal by several normality tests, another outlier detection method, the ROUT method is generally recommended.

The ROUT technique begins with a robust type of nonlinear regression, which is based on the assumption that the scatter follows a Lorentzian distribution. The data is then fit using the ROUT method. Once this is accomplished, an adaptive approach is used, which gradually gets more resilient as the procedure progresses. A modified version of the false discovery rate technique to handle multiple comparisons is used to identify outliers. Outliers are eliminated from the data, and the data is then analysed using a conventional least-squares regression. The ROUT technique is so named because it combines robust regression with outlier removal, and it is a combination of the two methods. If the data being analysed is simulated data with all scatter being Gaussian, the technique only finds (falsely) one or more outliers in around 1-3 per cent of tests. If the data being analysed contains one or more outliers, the ROUT technique performs exceptionally well at outlier detection, with an average False Discovery Rate of less than 1 per cent [20].

Normality test

Residuals from the pseudo-1st order model were subjected to four normality tests- Kolmogorov-Smirnov [21,22], Wilks-Shapiro [23], Anderson-Darling [24] and the D'Agostino-Pearson omnibus K2 test [25]. These tests require that the number of residual samples exceed at least ten. A lower number of samples tend to give inconclusive results [26,27]. Using graphical and numerical methods are two ways to search for normality. The simplest and easiest way to assess the normality of data is via graphical methods such as the normal quantile-quantile (Q-Q) plots, histograms or box plots [28]. The normality tests were carried out using the GraphPad Prism® software (Version 6.0, GraphPad Software, Inc., USA).

RESULTS AND DISCUSSION

Statistics often used in nonlinear regressions rely on the use of residual data, which is the difference between the expected and the actual values. Statistical analyses should be done to evaluate the adequacy of residues in randomness, do not include outliers, obey normality, and do not demonstrate autocorrelation. Usually, the greater the discrepancy between the expected and the observable values, the less well off the model [29]. Visual observation of the residual data (**Table 1**) shows nonconformity to normality. The normality tests also show the same conclusion with the residuals not passing all of the normality tests (**Table 2**).

The Grubbs' test deals with one aspect at a time while the ROUT method. Outliers are eliminated and test replicated before the test passes without revealing any outliers. As a general rule, sample sizes of 6 or less result in biased data sets. The residual of the fitted data of 15 points (**Table 1**) is generally large enough for further normality and Grubbs tests to be carried out. When doing nonlinear regression, the same assumption is made as to when performing linear regression: that the dispersion of data about the ideal curve follows either a Gaussian or normal distribution. Based on this assumption, the aim of regression is to minimise the sum of the squares of the vertical or Y-value distances between the points and the curve, which is well-known in mathematics. Outliers can have a significant impact on the sum-of-squares computation, resulting in misleading findings. We are not aware of any effective approach for finding outliers when fitting curves with nonlinear regression on a regular basis, though.

Table 1. Residuals from the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]).

Data Point No	Residual
1	1.084054
2	-5.18859
3	5.26507
4	-2.57351
5	0
6	-0.97973
7	0.394232
8	0.751616
9	-0.35637
10	-0.0926
11	-0.03283
12	-0.09837
13	-0.01385
14	-0.01128
15	0.038701

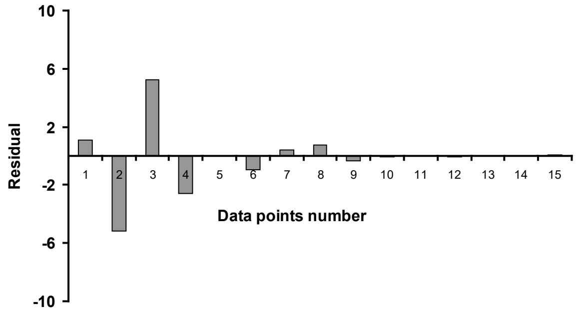

Fig. 1. Residual plot for the residuals from the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]).

Table 2. Normality tests of the residuals from the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]).

Test for normal distribution	
Anderson-Darling test	
A2*	1.484
P value	0.0005
Passed normality test (alpha=0.05)?	No
P value summary	***
D'Agostino & Pearson test	
K2	6.267
P value	0.0436
Passed normality test (alpha=0.05)?	No
P value summary	*
Shapiro-Wilk test	
W	0.8079
P value	0.0046
Passed normality test (alpha=0.05)?	No
P value summary	**
Kolmogorov-Smirnov test	
KS distance	0.2562
P value	0.0091
Passed normality test (alpha=0.05)?	No
P value summary	**
Number of values	15

The Grubb's test was applied to the residual results (**Table 3**). Grubbs test statistic defines the highest absolute variance from the survey mean in the sample standard deviation units. The critical value of *Z* from the statistical table for Grubbs' test for a single outlier using mean and SD was 2.548 (n=15). The Grubbs (Alpha = 0.05) ***g*** value was 2.518. As the *g* value was lower than the critical test statistics, then the statistics indicate an absence of an outlier. Individual Z value indicates that the residual with a value of -3 (row 3) was far from the rest but is not deem a significant outlier (p > 0.05).

Table 3. Descriptive statistics and calculated Z value for the residuals from the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoro-methylsulfonil) imide ([bmim][TF2N]).

Mean: -0.121
SD: 2.139
# of values: 15
Outlier detected? No
Significance level: 0.05 (two-sided)
Critical value of Z: 2.548

Row	Value	Z	Significant Outlier
1	1.084	0.563	
2	-5.189	2.369	
3	5.265	2.518	Furthest from the rest, but not a significant outlier (P > 0.05).
4	-2.574	1.147	
5	0.000	0.057	
6	-0.980	0.402	
7	0.394	0.241	
8	0.752	0.408	
9	-0.356	0.110	
10	-0.093	0.013	
11	-0.033	0.041	
12	-0.098	0.011	
13	-0.014	0.050	
14	-0.011	0.051	
15	0.039	0.075	

Despite the absence of an outlier, as demonstrated, the residuals did not pass all of the normality tests. When this occurs, the next recommended step is to use the ROUT method. The ROUT method indicates the presence of three outliers. After removal of the outlier, the model's normal probability or Q-Q plot was relatively straight (**Fig. 2**) and the model's residual passes two ot of the four normality tests (**Table 4**). The resulting histogram (**Fig. 3**) overlaid with the ensuing normal distribution curve reveals that the residuals roughly conforms to a normally distributed data and that the model used was adequately fitted.

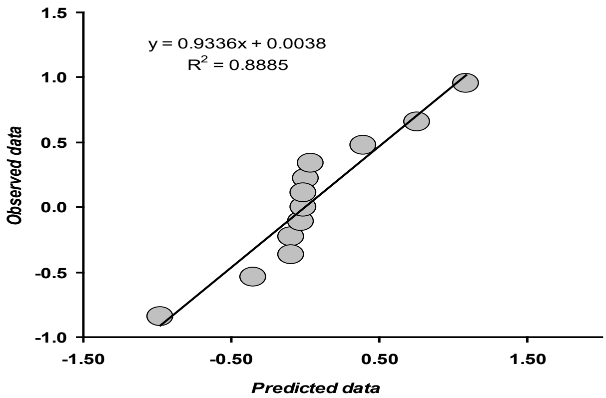

Fig 2. Normal Q-Q plot for the observed sample against theoretical quantiles for the residuals from the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]).

Table 4. Normality tests of the residuals from the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]) after removal of outlier.

Test for normal distribution	
Anderson-Darling test	
A2*	0.7155
P value	0.0451
Passed normality test (alpha=0.05)?	No
P value summary	*
D'Agostino & Pearson test	
K2	1.651
P value	0.4381
Passed normality test (alpha=0.05)?	Yes
P value summary	ns
Shapiro-Wilk test	
W	0.9048
P value	0.1829
Passed normality test (alpha=0.05)?	Yes
P value summary	ns
Kolmogorov-Smirnov test	
KS distance	0.2640
P value	0.0205
Passed normality test (alpha=0.05)?	No
P value summary	*
Number of values	12

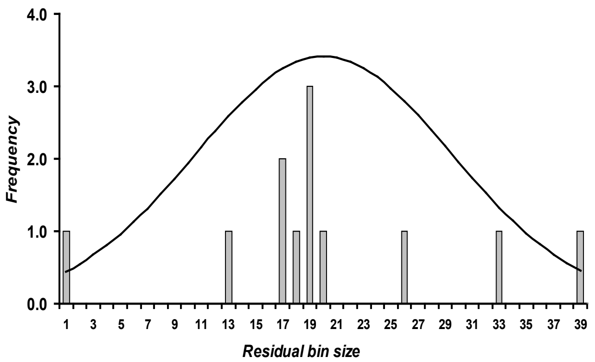

Fig. 3. Histogram of residual for the Carreau-Yasuda model in fitting the rheological behavior of the nonnewtonian fluid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonil) imide ([bmim][TF2N]) overlaid with a normal distribution (mean 0.057 and standard deviation 0.5191).

Although viscosity is frequently used in fluid mechanics to characterize a fluid's shear properties, it may not be adequate to represent non-Newtonian fluids in all circumstances. In order to better understand them, several additional rheological properties that relate to stress and strain rate tensors under a range of flow conditions, such as oscillatory shear or extensional flow, are used in conjunction with different measuring instruments known as rheometers. It is preferred to use tensor-valued constitutive equations, which are common in the field of continuum mechanics, to investigate the features of the system [15,30–33].

Shear thinning (as opposed to shear thickening) is a subset of non-Newtonian behavior of fluids whose viscosity decreases under shear strain. It is sometimes considered synonymous with pseudoplastic behaviour. Shear thinning is the most common type of non-Newtonian behavior of fluids and is seen in many industrial and everyday applications. Although shear thinning is generally not observed in pure liquids with low molecular mass or ideal solutions of small molecules like sucrose or sodium chloride, it is often observed in polymer solutions and molten polymers, as well as complex fluids and suspensions like ketchup, whipped cream, blood, paint, and nail polish [3,34–39].

Shear thinning fluids are a new class of fluids that have applications in polymerization and the production of multiple emulsions. Various models, such as the Williamson model [40], Ostwald-de-Waele model [1], the cross model [41], the Ellis model [1], the Carreu and the Carreau-Yasuda models [42,43] have been proposed to predict the rheological characteristics of shear thinning fluids.

The Cross and the Carreau-Yasuda models for non-Newtonian pseudoplastic fluids are two of the most widely used models for non-Newtonian pseudoplastic fluids. The Cross model is a non-Newtonian data-passive empirical equation with a variable coefficient [1]. This model (which describes asymptotic flow at zero (n_0) and infinite (n_∞) shear rates with no asymptotic viscosity and no yield stress) is more particular in that it is not a generalized model. Cross and Carreau-models Yasuda's may explain a wide range of fluid types, including dispersions, polymer melts, and polymer solutions, in two empirical equations, including dispersions and polymer melts. The Carreau-Yasuda model is another empirical equation that may be utilized with non-Newtonian data. Both the model Cross and the model Carreau-Yasuda have descriptions that are similar to one another [1,42,43]. In comparison to the Cross model, it contains more parameters and is the most often used variant of the fluid model of the power law. Emulsions, biopolymer solutions, protein solutions, polymer melts, and polymer solvents are all examples of fine fluids that may be described using the Carreau-Yasuda model for start-to-shear fine fluids. If the fittings for both models are near to their respective statistical function error analyses, the Cross model, which has lower parameters, should be selected as a rule of thumb. The Carreau-Yasuda model has found application in some biological systems, for instance in the modelling of the non-Newtonian blood flow in intracranial aneurysm cases, where the normality of the residuals has been checked with several normality models [4].

It is common to practise calculating the fitness of a mathematical model precisely through the use of residual measurements. As defined by a certain mathematical model, residuals are the difference between the sum expected and the total actually observed. The underlying premise is that the wider the disparity between the expected and observed values, the poorer the model is considered to be. A probable outlier is a data point that is out of the ordinary and that the researcher determines to be impossible based on a range of specific criteria. More specifically, an outlier in a study may be a unique characteristic that is far too uncommon in comparison to the rest of the population. To give an example, most outliers are only considered outliers if they are statistically excessively high for the distribution to the limit in the sample model, which is not always the case [44].

A simple strategy to identify potential outliers in testing is to include a boxplot, although more complex methodologies, such as the Chauvenet criteria in engineering and the 3-sigma criterion, coupled with the Z-score in chemometrics, are frequently employed. Despite the fact that these approaches are simple and quick, there is a considerably more effective way of employing the statistical test for outlier discovery than the methods described above. With the exception of one outlier, relevant assessments differ from the Dixon Q-test or the Grubbs ESD-test.

A variety of conditions have benefitted from the use of the Grubbs test to detect the presence of outliers [45–55]. The most significant restriction of the Grubbs test is that the thinking quantity of the outliers, denoted by the letter k, must be given explicitly. A failure to properly clarify the variable "k" can result in distorted results from the trials. A test called the Rosner Generalized Severe Studentized Deviate (ESD-test) is used when there are several outliers or when the exact number of outliers cannot be determined [56]. For example, if there is more than one outlier in a sample, the findings of the Grubbs test will be distorted, and when this occurs, the Ferguson sample skew test is more resistant to the misleading impact than the Grubbs test [57].

The number of bins and samples assessed determined the shape of the distribution. The W_2 statistic in the Wilks-Shapiro test is calculated using the anticipated values of the order statistics between identically distributed random variables as well as their independent covariance, as well as the regular normal distribution. The agreement is refused if the test statistics-W2 have a significant impact on the outcome. According to Royston, formalised euphemism is The Kolmogorov-Smirnov statistic, when applied to data, computes the cumulative residual frequency, which is a non-parametric numerical test [23]. It evaluates the link between the model and the observed values. It can also be used to compare two sets of data to see how they differ. The p value is derived using the difference between two combined distributions as well as the sample population size. On a more general level, the Central Limit Theorem (CLT) claims that as n approaches infinite (in actuality, n>30), the probability frequency distribution tends to fit the Gaussian distribution on any continuous variable (even for discrete variables such as Binomial or Poison distributions) [58,59]. The skewness and kurtosis of the distributions were analysed as a technique of quantifying the difference between the sample distributions and the usual distribution in order to determine the significance of the results in the D'Agostino-Pearson normality test method. Following that, the p-value of the sum of these inconsistencies or discrepancies is calculated. D'Agostino developed a variety of normality tests, the most extensively used of which is the omnibus K2 test [25]. More and more nonlinear regression curve fitting exercise works are reporting extensive testing for the normality of the residuals [60–68].

CONCLUSION

In conclusion, the normality checks performed on the residues used in this study revealed that the use of the the Carreau-Yasuda model in fitting the rheological behavior of the non-Newtonian fluid1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide ([emim][TF2N]) was initially not satisfactory due to the presence of three outliers. Removal of these outliers gave acceptable results in terms of normality tests and visual conformation of the residual, Q-Q plot and overlaid normality curve to the histogram, indicating that the model is now appropriate for the data. Much research on the use of the model utilised in the mathematical diagnostic of residues have been published, but it is commonly known that they have not gone any farther in their exploration. This may result in a data violation in the case of a Gaussian or regular distribution. The majority of nonlinear regression parametric prediction estimate methodologies rely on this assumption as a required but not sufficient condition. Methods such as the root mean square error, Pearson correlation coefficient (either standard or modified), the F-test, and the t-test are utilised on the basis of residuals that follow the normal distribution. If these assumptions were followed, Type I and Type II errors may be avoided. Furthermore, if diagnostic tests demonstrate that pollutants have breached any of the assumptions, the problem can be resolved in the field by adopting a variety of nonparametric treatments or changing the form of the therapies in question.

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