ESTIMATION RHEOLOGICAL PARAMETERS OF MANGO PULP (*MANGIFERA INDICA, L.*)

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***Abstract**. The rheological behavior of juices and pulps of fruits whose composition is primarily of water, besides the presence of various soluble and insoluble solids results from the interaction between these elements that contributes, in a potentialized or isolated way, when combined. Liquid products derived from fruit are biphasics systems, composed of solid particles dispersed in aqueous medium. The majority flow with pseudoplastics characteristics, showing sometimes, an initial resistance to the flow and/or a dependency of the time. The aim of this article is to present an inverse problem to estimation of rheological parameters of mango pulp (Mangifera Indica, L.). The rheological measurements were made at 10, 20, 30 and 40°C temperatures in rheometer Haake model VT550 with concentric cylinders systems attached to a thermostatic bath to control the temperature from samples. The equation used in the solution of the direct problem estimation of rheological parameters was the Mizhari-Berk equation with three parameters to determine: consistency Index (K), flow behavior index (n) and the square root of the initial stress of Mizrahi-Berk (Ko_M). The application of inverse method in the rheological characterization of mango pulp was effective in all temperatures which were studied. The aim of this work has been studying the rheological behavior of mango pulp solving a problem of parameters estimation through the minimization of the norm of ordinary least- squares using algorithm of the Levenberg-Marquardt*

***Keywords**: Inverse Method, Rheology, Pulp, Mango*

1. INTRODUCTION

The rheology of food is defined as the study of the deformation of raw materials, intermediate products and final products in food industry (Bourne, 2002). According to Costell *et al*. (1997) apud Toledo (2004), the importance of the study of rheology, besides strictly technological, is also economical and commercial, as the effects of transport and handling in the physical integrity of the food (both those that will be consumed fresh or that will be processed), its behavior during the manufacturing process and quality of the texture of the finished product depends largely on its response to external forces.

The rheology plays an important role in developing, manufacturing and processing of food products. According to Bourne (2002), the flow properties and deformation of food are important in the design of equipment such as pumps, pipes, belts, spray devices, etc., to obtain information about the structure of foods or on the conformation of the molecular constituents of them, especially the macromolecular constituents, and still to make measures that will help in the assessment of sensory attributes related to the texture of the product. Based on these measurements, the process and/or a formulation for a given product may be changed in order to improve the final product taking into account the textural parameters desirable to consumers.

According Costell and Durán (1982) apud Ferreira *et al*. (2002), all liquid products derived from fruit are biphasics systems, composed of solid particles dispersed in aqueous medium. Some of them have Newtonian behavior, although most flow with pseudoplastics characteristics, showing sometimes an initial resistance to flow and/or a dependency of time.

There are some mathematical models which were developed empirically to represent the rheological behavior of non-Newtonian fluids. For pulp of fruit with pseudoplastic behavior, the most used are the mathematical models of Herschel-Bulkley, Mizrahi-Berk and Ostwald-of-Waelle.

There are few studies involving application of inverse methods in the rheology of food. Some authors used inverse methods to estimate thermophysical properties of food in processes of heat transfer. Silva *et al*. (2006), applied inverse methods in determining the mass diffusivity of cherry tomatoes subjected to an osmotic dehydration; Silva (2007) applied inverse methods to estimate the mass effective diffusivity of mushroom of the species *Agaricus Blazei* subjected a process of air convective drying; Mendonça, Filho and Silva (2004) used inverse methods in determining the thermal conductivity and volumetric thermal capacity of spherical fruits in transient process; Monteau (2007) used inverse methods in determining the thermal conductivity of sandwiche's bread.

The estimation of parameters can be made through direct methods or inverse methods. The direct methods consist in design an experience so that the measured values can be expressed through a mathematical function. The mathematical function should express the properties directly in terms of the measures taken. Thus, each experience will allow the calculation of the value of a single parameter. The use of inverse methods is more advantageous than the use of direct methods therefore allow to estimate the largest possible number of parameters from a simple experiment, increasing the accuracy, combine several experiments to estimate the same parameters and determine the limits of confidence (Beck and Arnold, 1977). An inverse problem determines unknown causes based on observation of its effects. In direct problems whose solution can be obtained by analytical methods or numeric the opposite occurs, the solution of the problem represents the effect based on full description of their causes. The inverse problems are mathematically classified as "ill-posed", that is, may have solution or not, and if exist solution that may be single or stable. The main feature of this type of approach is to obtain the solution of the physical problem of the indirect way (Guimarães, 2007).

The aim of this work has been to study the rheological behavior of mango pulp solving a problem of estimation of parameters through the minimization of the norm of ordinary least-squares using algorithm of the Levenberg-Marquardt.

2. MATERIALS AND METHODS

The mango pulp was obtained in its frozen form. It was used a Haake rheometer model VT550 with concentric cylinders system equipped with a thermostatic bath to control the temperature of the samples. Five replicates were made for each temperature of 10, 20, 30 and 40°C. The range of shear rate was from 0.1 to 400 S^{-1} resulting in 40 points of deformation for each studied temperature, and the points of deformation collected every 30 seconds.

2.1. Direct Problem

Some of the mathematical models more used in determining of rheological parameters of non-Newtonian fluids are the power-law equation, Bingham equation and Mizrahi-Berk equation, shown in Eq. (1), (2) and (3), respectively:

$$\tau = K\gamma^{n} \tag{1}$$

$$\tau = \tau_0 - \mu_p \gamma \tag{2}$$

$$\tau^{0,5} = K_{0M} + K\gamma^{n} \tag{3}$$

Where τ is the shear stress, τ_0 is the initial stress, K is the consistency index, n is the flow behavior index, γ is the shear rate, K_{0M} is the square root of the initial stress of Mizrahi-Berk and μ_p is the plastic viscosity.

The model proposed by Mizrahi and Berk (1972) is the best that describe the rheological behavior of pulp, fruit juices and purees, as the authors developed based on the model of a suspension of particles interacting in a pseudoplastic solvent (Pelegrine, 2000); for this reason we choose the equation as the direct model in estimation of the rheological parameters of mango pulp, using inverse methods.

2.2. Sensitivity Study

In the estimation of parameters through inverse methods is extremely important to analyze the sensitivity coefficients. A sensitivity coefficient is the first derivative of variable according to the unknown parameter. Let be $\tau(\gamma, \beta)$ the shear stress, $\beta = (\beta_1, \beta_2, \beta_3)$ the unknown parameters of Mizrahi-Berk model, and γ the shear rate. The sensitivity coefficient to the variable τ is given by:

$$X_j(\gamma, \beta) = \frac{\partial \tau}{\partial \beta_j} \tag{4}$$

The sensitivity coefficient represents the variation of the variable of state due to a change in value of the unknown parameter. It also expresses how the model responds to a small change of the parameters and allows measure the importance of this effect on the performance of the studied variable (Beck and Arnold, 1977).

2.3. Inverse Problem

At the solution of the inverse problem, the information is extracted from experimental data. The experiences with mango pulp provided data of shear stress like function of shear rate, resulting in a total of 40 points for each studied temperature.

According to Silva *et al* (2006), methods of estimation of parameters involve various techniques of approximation. A criterion for approximation is a quadratic function composed of one or two objective functions plus a Bayesian function.

$$S(\beta) = [Y - X(\beta)]^T W [Y - X(\beta)] + [\mu - \beta]^T U [\mu - \beta] \tag{5}$$

Where Y (nx1) is the array of measures, W (nxn) and U (pxp) are matrices that depend on the type of estimator. For Ordinary Least Squares (OLS) W = I and U = 0, resulting:

$$S(\beta) = [Y - X(\beta)]^T [Y - X(\beta)] \tag{6}$$

There are many methods for minimizing or maximizing a non-linear function. The choice depends on the number of parameters to be estimated and to some extent the structure of the criterion. For non-linear problems, the method of Gauss-Newton is the most appropriate when the number of unknown parameters is not great.

For non-linear problems, from the viewpoint of estimation of parameters, an iterative procedure should be used. The Levenberg-Marquardt method was considered to minimize the norm of ordinary least squares shown in the Eq. (6).

2.3.1. Levenberg-Marquardt Method

The Levenberg-Marquardt (Press, *et al*., 1989) method provides a numerical solution to the problem of minimization of a function, generally non-linear, on a space of parameters for this function. It is a curves-fitting algorithm very used. Its main application is in the least squares curve fitting problem. Given a set of empirical data pairs of independent and dependent variables (x_i, y_i) the aim is the optimization of the parameters β of model curve $f(x,\beta)$ so that the sum of the squares of the deviations $S(\beta)$ becomes minimal.

$$S(\beta) = \sum_{i=1}^{m} [y_i - f(x_i,\beta)]^2 \tag{7}$$

The method introduces a restriction on the criterion of minimization to overcome the instability of the method of Gauss-Newton. Based on the criterion of ordinary least squares, the iterative formula has the following expression:

$$\beta^{(k+1)} = \beta^{(k)} + \left[J^{T(k)} J^{(k)} + \lambda^{(k)} \Omega_m{}^{(k)}\right]^{-1} J^{T(k)} \left(Y - X^{(k)}(\beta)\right) \tag{8}$$

Where $\lambda^{(k)}$ a non-negative number is called damping factor, $\Omega_m{}^{(k)}$ is a diagonal matrix of positive terms and $J^{(k)}$ is the sensitivity coefficients matrix.

The intention of the term $\lambda^{(k)}\Omega_m{}^{(k)}$ in the Eq. (8) is dampen fluctuations and instability occurred because of character of conditioning bad the problem. This avoids that the matrix $J^T J$ be non-singular at the beginning of the iterative procedure and Levenberg-Marquardt method tends to the steepest descent method. The parameter $\lambda^{(k)}$ is then reduced slowly as the process of iteration moves towards to the solution the problem of parameter estimation and then the Levenberg-Marquardt method tends to the Gauss method (Mejias *et al*, 1999).

Like other numeric minimization algorithms, the Levenberg-Marquardt algorithm is an iterative procedure. To start a minimization of the function it is necessary an initial guess for the vector of parameters β. The procedure iterative starts with an initial guess, $\beta^{(0)}$, in each step the vector β is modified until:

$$\frac{\left|\beta_i^{(k+1)} - \beta_i^{(k)}\right|}{\left|\beta_i^{(k)}\right| + \xi} < \delta \text{, para i = 1, 2, ..., p} \tag{9}$$

Where δ is a small number as 10^{-3} which representing the relative error of convergence and ξ ($<10^{-10}$) avoid the situation where $\beta_i^{(k)} = 0$.

Initially we choose a low value to $\lambda^{(k)}$, $\lambda^{(0)}$ = 0.001 and so the minimization process follows the steps below:

Step 1: Solve the direct problem through of Mizhari-Berk model with the available estimate parameter $\beta^{(k)}$.

Step 2: compute $S(\beta^k)$ by Eq. (6)

Step 3: compute the sensitivity matrix $J^{(k)}$ and then the matrix $\Omega_m{}^{(k)}$

Step 4: compute the increments for the unknown parameters by using Eq. (9)

Step 5: compute the new estimate β^{k+1} as $\beta^{k+1} = \beta^k + \Delta\beta^k$

Step 6: solve now the direct problem with the new estimate β^{k+1} in order to find $X(\beta^{k+1})$. Then compute $S(\beta^{k+1})$, as defined by Eq. (6)

Step 7: if $S(\beta^{k+1}) \geq S(\beta^k)$, replace $\lambda^{(k)}$ by 10 $\lambda^{(k)}$ and return to step 4.

Step 8: if $S(\beta^{k+1}) < S(\beta^k)$ accept the new estimate β^{k+1} and replace $\lambda^{(k)}$ by 0.1 $\lambda^{(k)}$

Step 9: check the stopping criteria given by Eq. (9).

The subroutines Mrqmin.for from Numerical Recipes was used in this paper.

3. RESULTS AND DISCUSSION

The direct problem consists to obtain the shear stress as function of shear rate, using the estimated parameters, through Mizrahi-Berk model. This model was obtained by a modification of the Casson equation and originally was applied in rheological characterization from concentrate orange juice. The Mizrahi-Berk model has three unknown parameters: consistency index (*K*), flow behavior index (*n*) and the square root of the initial stress of Mizrahi-Berk (Ko_M). The flow behavior index indicates how different the fluid is from the Newtonian model. The value of the consistency index indicates the degree of resistance of fluid before the flow. The greater the value of *K*, more 'consistent' the fluid is (Machado, 2002). The third parameter is related to the residual tension of the fluid. The physical behavior of fluids with residual tension is usually explained in terms of its internal structure, which is capable of difficult the movement if the values of shear stress are smaller than the residual stress (Barnes et al., 1989 apud Sato, 2005).

3.1 Identificability

The viability analysis of parameters simultaneous estimation in a single experience is part of the problem identificability. According to Beck and Arnold (1977), there are cases where the parameters can not be estimated simultaneously in a single experiment. There is impossibility to estimate the parameters simultaneously in the following cases: Some parameters can only be estimated at a certain interval of the dependent variable or in certain values; If one or more parameters have sensitivity coefficients two orders of magnitude smaller than the other, it will be difficult to estimate precisely these parameters; If there is a linear dependence between the parameters of the model will not be possible the estimation from them simultaneously.

The figures (1) to (4) show the normalized sensitivity coefficients of the parameters to the model in the temperatures of 10, 20, 30 and 40°C respectively. According to visual analysis of the fig. (1) to (4) it was observed that there is probably a linear dependence between the consistency index (*K*) and the behavior index from fluid (*n*) since the behavior of the curves are very similar showing proportionality between them. This is one of the reasons for impossibility of simultaneous estimation of these two parameters. Problems due to a bad-conditioning in the matrix $\left|J^T J\right|$ into Eq. (8) and small magnitudes of the sensitivity coefficients are other important reasons that prevent the correct estimation of the parameters generating great uncertainties in the estimation of them. It was found that the rheological parameter with greater sensitivity to the mathematical model was the behavior index (*n*), meaning that its estimate by inverse methods is easier to obtain. On the other hand, the parameter that presented smaller sensitivity to the model was the square root initial stress Mizrahi-Berk model (K_{OM}), meaning that this parameter is difficult to estimate by inverse methods. According to Pelegrine *et al* (2000), the experimental determination of the initial stress is very difficult to be obtained in simple rheometer and generally the value of K_{OM}, which depends on the initial stress, it is only the result of an fitting of model the experimental points.

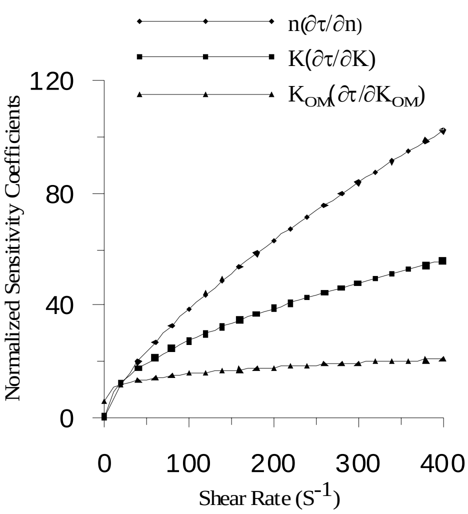

Figure 1: Sensitivity Coefficient in Temperature of 10ºC

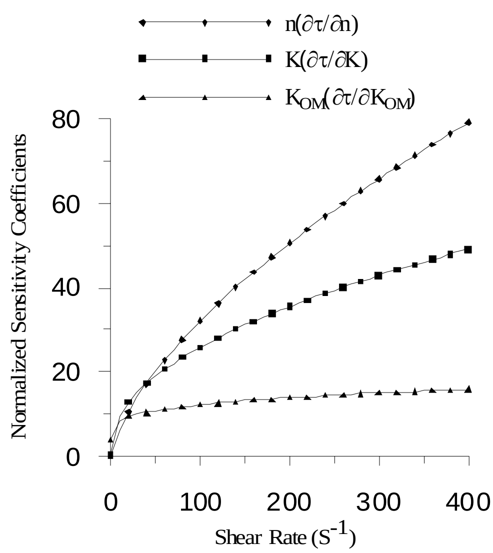

Figure 2: Sensitivity Coefficient in Temperature of 20ºC

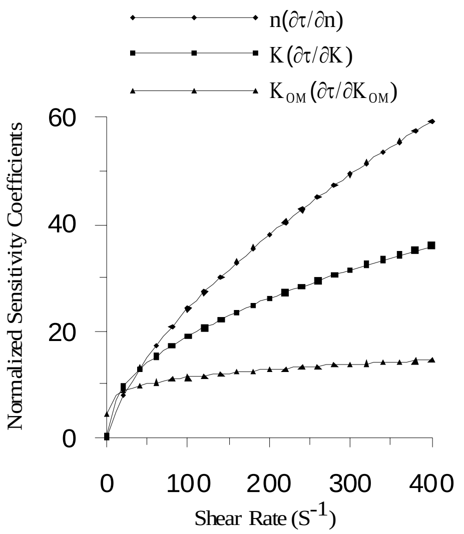

Figure 3: Sensitivity Coefficient in Temperature of 30ºC

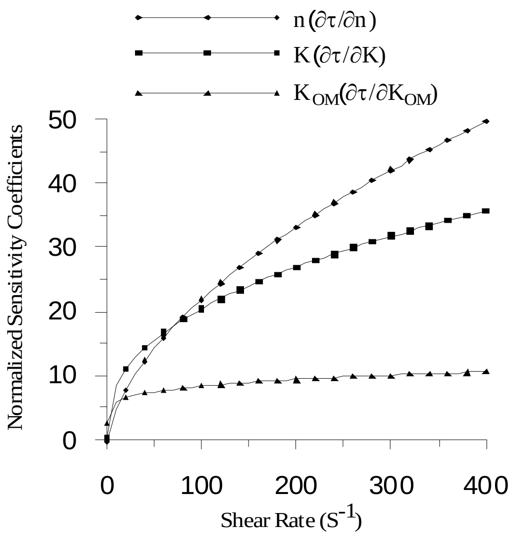

Figure 4: Sensitivity Coefficient in Temperature of 40ºC

3.2 – Parameters Estimation

The table (1) shows the values of the Mizhari-Berk model parameters to mango pulp which were estimated by inverse methods. The values of the square root of the initial stress of Mizrahi-Berk (K_{oM}), consistency index (K) and flow behavior index (n) are shown to a temperature range of 10 – 40^{o}C.

Due to a linear dependence between the consistency index (K) and the behavior index of the fluid (n) conclusive by analysis of the fig. (1) to (4), there is impossibility of simultaneous estimation of these two parameters, therefore, the value of the behavior index of the fluid (n) was considered known, according to values found by Bezerra, Queiroz and Gasparetto (2001).

Table 1: Rheological Parameters Estimated by Inverse Methods

Parameters of the Mizrahi-Berk Model Estimated by Levenberg-Marquardt Method				
T(ºC)	*10*	*20*	*30*	*40*
K_{OM} *(Pa)*	1.4732 ± 0.0005	2.0801 ± 0.0004	2.1861 ± 0.0003	2.4795 ± 0.0003
K (Pa.s)	0.8219 ± 0.0001	0.4456 ± 0.0001	0.2618 ± 0.0001	0.1353 ± 0.0001

Some divergences are noticed within the values of the table above and the values found by Bezerra, Queiroz e Gasparetto (2001). This can be due to the differences in the composition of mango pulp used in the experiments such as differences in the soluble solids content, which influence in the rheology of the fruits pulps. The Levenberg-Marquardt method considers the confidence of each experimental point in the estimation and therefore it allows determining the parameters confidence interval, which assures greater reliability in the results. The figures (5) to (8) show the behavior of the flow curves experimentally obtained and flow curves obtained by direct problem using the parameters estimated through Levenberg-Marquardt method at temperatures of 10, 20, 30, 40ºC for mango pulp. The plot shows similarity in the behavior of the experimental and theoretical curves which guarantees the quality of the methodology applied. The graphics were built with the shear stress (τ) in function shear rate (γ).

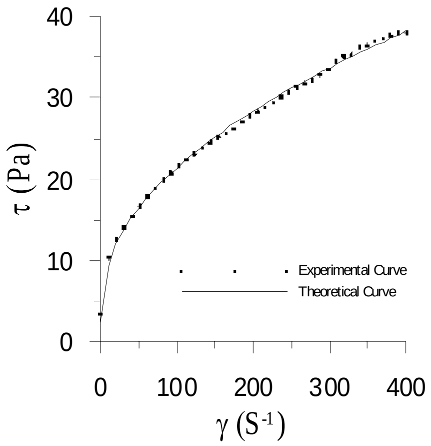

Figure 5: Experimental and Theoretical Curves in Temperature of 10ºC

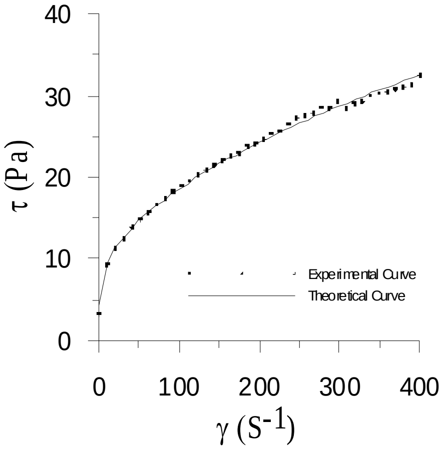

Figure 6: Experimental and Theoretical Curves in Temperature of 20ºC

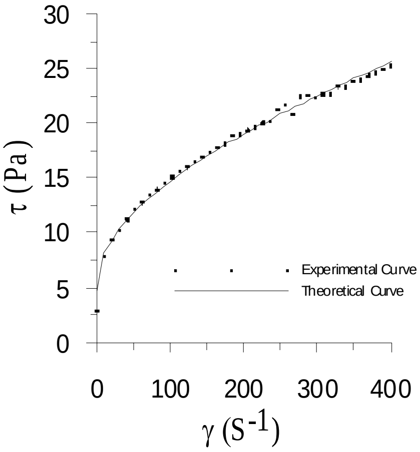

Figure 7: Experimental and Theoretical Curves in Temperature of 30ºC

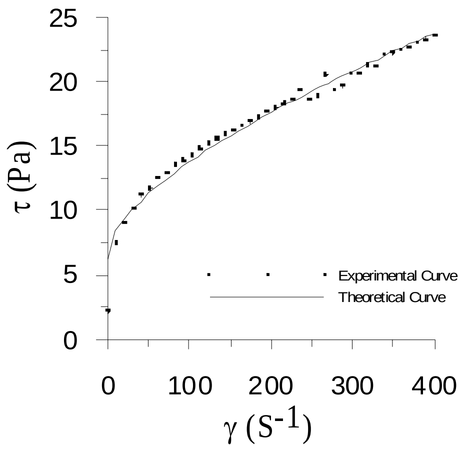

Figure 8: Experimental and Theoretical Curves in Temperature of 40ºC

After the estimation of the rheological parameters by using the Levenberg-Marquardt iterative method, the direct problem was resolved and the curves obtained in the studied temperatures were confronted with the experimental curves, as shown in Fig. (5) to (8). By observing these figures, it is checked that the theoretical points are very close to the experimental points and the uncertainties in the estimation of the parameters are very small. This indicates that the parameters of the Mizhari-Berk model were correctly estimated by used method.

The consistency of the method of optimization presented in this paper can also be confirmed by the analysis of the residues. The residues shown in the figures (9) to (12) are the difference between the experimental shear stress and shear stress computed by using estimated parameters. Through analysis of the residues obtained in the studied temperatures, it is noticed the quality of the applied methodology due points around from zero, with a random relatively distribution which indicates that the proposed method has shown effectiveness for estimation of rheological parameters.

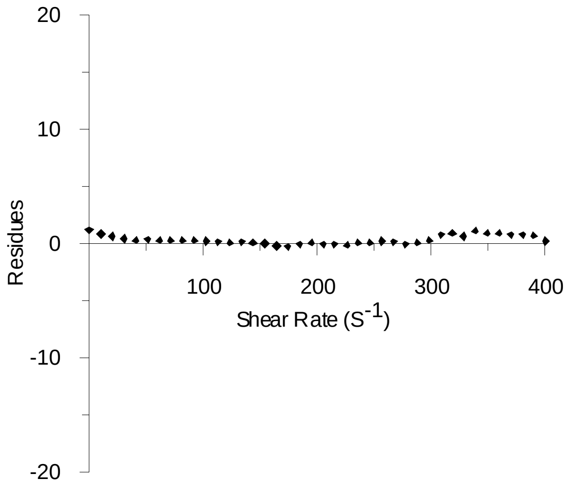

Figure 9: Computed Residues in Temperature of 10ºC

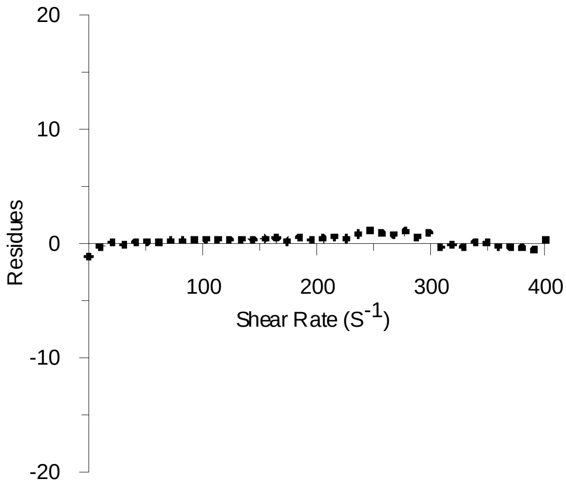

Figure 10: Computed Residues in Temperature of 20ºC

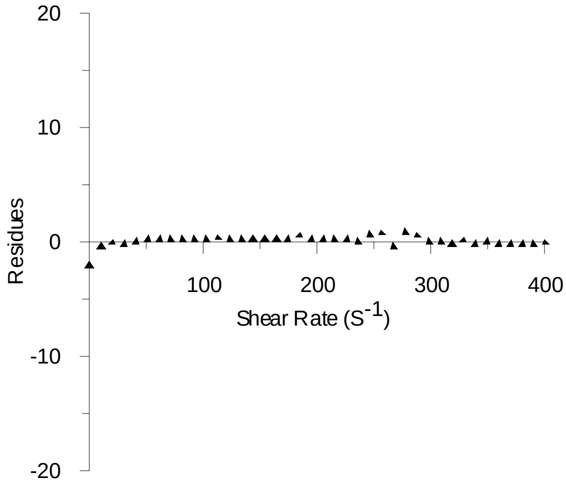

Figure 11: Computed Residues in Temperature of 30ºC

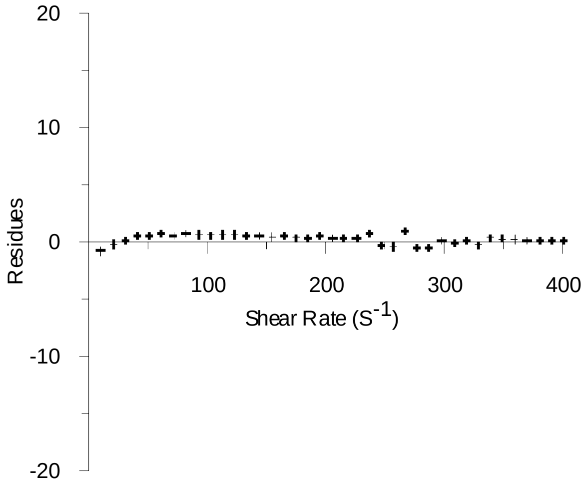

Figure 12: Computed Residues in Temperature of 40ºC

4. CONCLUSION

An inverse problem was solved to estimate rheological parameters of mango pulp. The Mizrahi-Berk mathematical model was used to solve the direct problem and the non-linear problem of minimization of the quadratic differences that allows identifying the parameters, was resolved by Levenberg-Marquardt iterative method. It was noticed through analyses of the sensitivity coefficients that consistency index (*K*) and behavior index (*n*) could not be estimated simultaneously. This study allowed identifying the parameters that would be possible to estimate.

The inverse methods when compared with statistical methods have the advantage of providing the additional confidence interval of the identified parameters.

The application of inverse methods in the rheological characterization of mango pulp was satisfactory in all studied temperatures.

5. ACKNOWLEDGEMENTS

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