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Statistical Analysis of Measured Sucrose Solubility, in Complex System and Its Prediction with UPDH Model

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Abstract. The sucrose solubility was measured at atmospheric pressure, for complex system, at temperature equal to 70°C. The complex system was assimilated to industrial sugar juices characterized by four pseudocomponents impurities; those are invert sugar, amino acid, carboxylic acid and ash. The variation intervals of these impurities were chosen proportionately small as in industrial sugar juices. The analysis of the experimental results, carried out using the Pareto diagram and the ANOVA test, shows the main effect of the composition of reducing sugar, ash and water with a contribution of 90% of total effects on the sucrose solubility and a correlation coefficient of 98%, without any interaction effect between the different variables studied. The UNIFAC Pitzer Debye-Hükel (UPDH) model was used to predict experimental sucrose solubility, in complex system. Some binary interaction parameters available in the literature were exploited and new others were estimated in this work. The model describes the experimental sucrose solubility in complex system, with mean absolute error of 3 % and maximum error of 7 %.

Keywords: Experiments, Sucrose solubility, Industrial sugar juice, Prediction, UPDH model.

1. Introduction

The Solubility of sucrose is very important both from an academic and industrial point of view. It has been applicated in different fields of industry [1]. Increasingly, modern process design involves the use of computer-aided process design techniques. The abundant literature concerning the prediction of this property for sugar did not cease to increase especially with the development and overgrowing demand from these industries. Despite the fact that the methods developed are numerous and diverse [2], empirical methods and those such Margule, UNIQUAC and UNIFAC are still the more usual answers to this demand. The classical UNIFAC model gives bad prediction of sucrose solubility in sucrose-water and in sucrose-glucose-fructose-water systems. The

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effect of making temperature-dependent binary interaction parameters between groups gives a strong reduction in the mean absolute error for the binary system, but poor results are obtained for the quaternary system. Introducing two new main groups, pyranose ring and furanose ring [3], UNIFAC model gives satisfactory prediction of sucrose solubility in sucrose-glucose-water and sucrose-fructose-water systems. Good estimations are obtained to predict solubility for binary systems: sucrose-water, glucose-water and fructose-water using UNIQUAC model. The latter is applied with a new equation, which requires dilution enthalpy. Peres and Macedo [4] use the same approach, by extending its application to the ternary systems. The same systems are successfully studied by Spiliotis and Tassioss [5], using UNIFAC. This method is applied to industrial sugar of fruit juices and synthetic honey, considered as sugar aqueous solution. In their investigation, application for industrial sugar is limited to predict water activity property. Van der Sman [6] have shown the theory reliability of the Flory-Huggins, to predict sucrose solubility in water, and two solutes solubility in water system containing two carbohydrates for different concentrations and temperatures. Crestani et al. [7] carried out an experimental study relating to the measurement of sucrose solubility in water, for purities equal to 0.996, 0.958 and 0.917 and for temperatures of 10 to 100 °C. The main impurities considered in their solutions are glucose and fructose. The data obtained were exploited, to show the poor prediction by UNIQUAC, and UNIFAC models, for the studied system. To improve these results, they modified the Nývlt equation and its application made it possible to predict the solubility of sucrose in the systems studied with an average error of 2.54%. Literature analysis shows that there has been much work on obtaining g^E models for describing and correlating phase equilibrium, but no method based on coupling UNIFAC to Pitzer Debye–Hückel (UPDH) approach exists for rigorous sucrose solubility prediction in industrial sugar. Particular attention is given here to examine UPDH method for solubility property of sucrose in industrial sugar solutions assimilate to complex system. The latter is characterized by a mixture of some impurities (invert sugar, amino acid, carboxylic acid and ash) on sucrose solubility. An experimental study was carried out to measure sucrose solubility in complex system. The obtained results were analyzed statistically before being used for the validation of the developed model.

2. Industrial sugar juices characterization

Sucrose is generally processed from cane and beet sugar. The quality of this product is influenced by the presence of some impurities during the different steps of the process [8]. Glucose and fructose, called reducing sugar, are present in both cane and beet juice; in addition, another quantity comes from sucrose inversion, under the effect of temperature and pH [9]. This invert sugar also affects industrial production of sugar cane, producing a browning effect and minimizing sucrose content [10]. The dextran that is a present in sugarcane has no effect on the sucrose solubility, but it decreases the growth rate of sucrose [11]. Its biodegradation decreases the time of crystallization, resulting in decreases of the production costs, in sugar manufacture [12]. At high temperatures during juice purification, reducing sugars break down to produce different acids. Unwanted dark colored products can also form in the juice by Maillard reaction between reducing sugars and amino acids present in the sugar juice [13]. There are many amino acids identified in beet sugar as given in reference [14]. The quantitative analysis of organic acids in the juice of sugar cane processing has identified several carboxylic acids [15]. Also, different phenolic acids are identified in sugarcane and are color precursors in cane sugar manufacture [16]; Gallic acid exists in large quantities in this juice [17]. The detection of some of these organic acids and inorganic anions in sugars can be an important criterion for the quality of the sugars marketed. According to Messnaoui et al. [18] the NaCl presence increases the sucrose solubility in water, when they studied a ternary NaCl-sucrose-water system, for sucrose concentration varying from 0.5 to 5.5 mol·kg−1 in the NaCl molality from 0.5 to 6.0 mol·kg−1 at 298.15 K. The same effect was seen when studying the KCl-D–sucrose-water system [19].

Bibliographic analysis shows that industrial juice extracted from sugar cane or beet is very complex system characterized by different constituents. Each type of these constituents is present in industrial sugar juice as impurities and can have a more or less important effect on the thermodynamic properties predictions. Usually, they are calculated by likening industrial juice to a simple ternary system consisting of sugar-non sugar and water. Almost all methods existing in the literature to study the industrial sugar processes are based on this ternary system. It would be more interesting to represent the industrial juice by more than three pseudocomponent. This is an approach that is adopted successfully, in our previous work [20]. So, the main impurities of sugar factories were considered, those are invert sugar, amino acid, carboxylic acid and mineral matter and assumed as four pseudocomponents. So, the beet or cane sugar juice was considered as sucrose-invert sugar-amino acid-ash-water system. This system considers some phenomena like degradation reactions, specific effects of some impurities on sucrose solubility.

3. Methodology

3.1. Experimental work

The experimental samples of complex system were realized according Box-Wilson experimental design [21]. The latter limits the number of experiments required and permits to test the interaction between different variables by changing them simultaneously. Four variables, which are pseudocomponents compositions were considered; thus 25 experiments were realised. Each pseudocomponent was represented by one or two main pure substances [20]: glucose and fructose for invert sugar pseudocomponent; aspartic acid for amino acid pseudocomponent; citric acid for carboxylic acid pseudocomponent; and NaOH and KCl for ash pseudocomponent. The contents of these impurities were chosen proportionately low as in industrial sugar juices. The composition of fructose and glucose was varied from 2 to 4 %; for amino acid, amounts of aspartic acid was used varying from 0.02 to 0.5 %; for carboxylic acid, citric acid was varied from 0.3 to 1% and for ash, the concentration interval was from 0.7 to 2 %. The sucrose solubility in studied complex system was obtained using the same isothermal method followed by Abed et al. [3]. The method principle is based on dissolving an excess of solid solutes and maintaining it under reaching equilibrium, at constant temperature. Then, the concentration of the solutions is measured. The procedure followed consists to introduce the samples into 10-100 cm^3 Erlenmeyer flasks with caps and are placed in a water bath having a vibration device. The water bath temperature set at 70 ° C, having 0.02 °C accuracy. The SLE is checked almost every day, by measuring the refractive index of a solution taken with Pasteur pipettes. When the variation of this index becomes negligible, the SLE is assumed to be reached. The later was achieved after almost twenty days of intimate contact and during this period the viscosity of the solution gradually increased. By allowing the samples to stand for more than 1 h at constant temperature, the crystals were separated by settling and made up by filtration due to the viscosity of the solutions. The accuracy of the method was evaluated in terms of reproducibility. The analysis methods of sugar industry were carried out by the same techniques followed by abed et al. [3].

3.2. Thermodynamic model

The thermodynamic equilibrium between two phases, Solid (S) and Liquid (L), is given for each component, i by this equation:

$$f_i^S = f_i^L \tag{1}$$

The solid fugacity in the liquid phase is given by the following equation:

$$f_i^L = \gamma_i x_i f_i^{\circ L} \tag{2}$$

Where: x, the solute mole fraction in the solution and $f_i^{\circ L}$, the pure solute fugacity in a subcooled liquid state below its melting point. When the solvent does not enter the solid phase, the solid solute fugacity equals to that of pure solid:

$$f_i^S = f_i^{\circ S} \tag{3}$$

Taking equation (3) and (4) in equation (2), the solid solubility in liquid at atmospheric pressure can be calculated by this equation:

$$\mathrm{Ln}(\gamma_i x_i) = \frac{\Delta H_m}{R}\left(\frac{1}{T_m} - \frac{1}{T}\right) \tag{4}$$

Where: ΔHm (J/mol), the enthalpy of melting and Tm (K), melting temperature.

The thermodynamic model for the sucrose solubility calculation in the complex system consists to combine UNIFAC method given by [22], with the Debye–Hückel theory [23]. This combination makes it possible to give the predominant short and long range interactions between the components in solution. Hence, the UPDH method is obtained by two terms:

$$Ln\gamma_i^{UPDH} = Ln\gamma_i^{PDH} + Ln\gamma_i^{UNIFAC} \tag{5}$$

First, concerned the Debye-Hückel term [23], which accounts for long-range forces. Its expression in this contribution is obtained by this equation:

$$Ln\,\gamma_i^{PDH} = -\left(\frac{1000}{M_s}\right)^{\frac{1}{2}} A_\phi \left[\frac{2 z_i^2}{\rho} Ln\left(1 + \rho I_X^{\frac{1}{2}}\right) + \frac{z_i^2 I_X^{\frac{1}{2}} - 2 I_X^{\frac{3}{2}}}{1 + \rho I_X^{\frac{1}{2}}}\right] \tag{6}$$

Where :

$$A_\phi = \frac{1}{3}\left(\frac{2\pi N_A d_s}{1000}\right)\left(\frac{e^2}{D_S K T}\right)^{\frac{3}{2}} \tag{7}$$

Where: M_s, solvent molar weight (g/mol); N_A, Avogadro number (mol^{-1}); d_s, solvent density (g/cm^3): e, electron charge ($4.802654\ 10^{-10}$eu); K, Boltzman constant ($1.38048\ 10^{-16}$erg K^1); Ds, solvent dielectric constant; I_x, ionic strength in mol fraction scale; ρ, minimum distance parameter fixed at 14.9 by Pitzer. z_i, charges number of ions i.

The second part is a non-electrolyte UNIFAC model, allowing to calculate the activity coefficient of group, by two terms [23]:

$$Ln\gamma_i^{UNIFAC} = Ln\gamma_i^C + Ln_i^R \tag{8}$$

The first term of contribution is etermined as follow:

$$Ln\gamma_i^C = Ln\frac{\Phi_i}{x_i} + \frac{z}{2}q_i Ln\frac{\theta_i}{\Phi_i} + l_i - \frac{\Phi_i}{x_i}\sum_j x_j l_j \tag{9}$$

$$l_i = (\frac{z}{2})(r_i - q_i) - (r_i - 1) \tag{10}$$

where, z is equal to 10, x_i the mole fraction of compound i, in the liquid phase, r_i and q_i are the volume and surface area parameters of compound i.

$$\theta_i = \frac{x_i q_i}{\sum_j x_j q_j} \quad \text{and} \quad \Phi_i = \frac{x_i r_i}{\sum_j x_j r_j} \tag{11}$$

$q_i = \sum \nu_k^i Q_k$, area parameter; $r_i = \sum \nu_k^i R_k$, parameter of volume and υ_k^i, the number of functional group k in species i. Q_k and R_k are combinatorial part of the pure component area and volume parameters, respectively, for group k.

The residual part is given by the solution-of-groups concept, expressed by:

$$Ln\ \gamma_i^R = \sum_k \upsilon_k^i (Ln\ \Gamma_k - Ln\ \Gamma_k^i) \tag{12}$$

Where: Γ_k, and $\Gamma_k^{(i)}$ are the group residual activity coefficient that for a reference solution of pure *i*, respectively; these two parameters are given by:

$$Ln\Gamma_k = Q_k\left(1 - Ln(\sum_m \theta_m \tau_{mk}) - \sum_m (\frac{\theta_m \tau_{km}}{\sum_n \theta_n \tau_{nm}})\right) \tag{13}$$

Where: $\theta_m = \frac{X_m Q_m}{\sum_n X_n Q_n}$ and $X_m = \frac{\sum_j x_j \upsilon_m^j}{\sum_n \sum_j x_j \upsilon_n^j}$

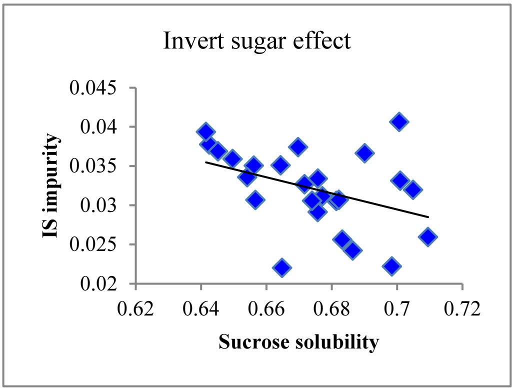
$$\tau_{mn} = exp(\frac{-a_{mn}}{T})$$

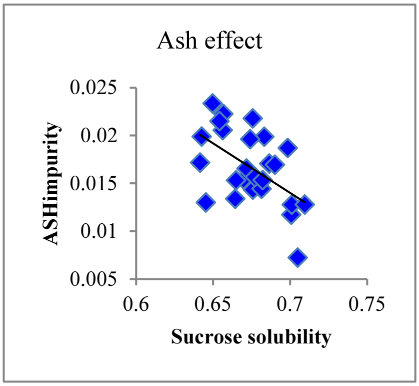
a_{mn} is the interaction parameter, for main groups m and n.

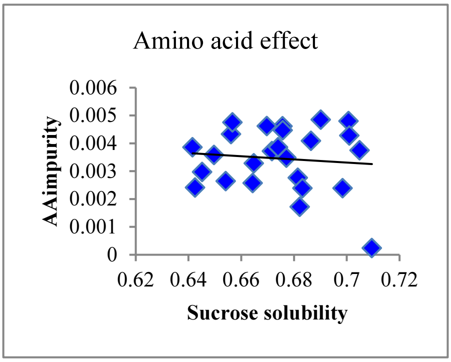
4. Results and discussion

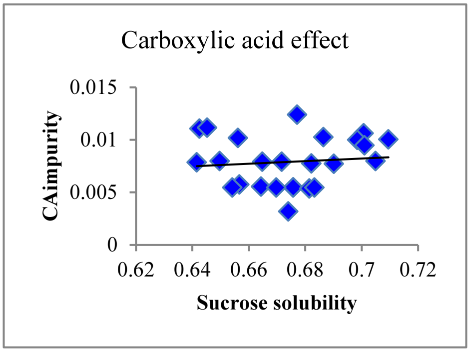
4.1. Experimental results

The results found in this investigation are obtained with experimental error that not exceeds 1%. These results of the complex system, for which the pseudocomponents have been associated in varying proportions are analyzed statistically. This analysis was performed using effect plot and Pareto chart as well as the ANOVA test [24]. Figure 1 shows experimental results of each pseudocomponent effect on sucrose solubility, in complex system at 70°C. The main effects observed in this figure are due to invert sugar and ash, showing a negative correlation between sucrose solubility and each of these two pseudocomponent. A decrease in the mass fractions of each of reducing sugars and ash resulted in a corresponding increase in the sucrose solubility. This can be explained by the salting-out effect of these two impurities as demonstrated, by Kelly on sucrose-glucose-fructose-KCl system [25]. The less significant effect on the sucrose solubility is obtained for the carboxylic acid and amino acid impurities (Figure 1). In the literature it has been reported that the amino acid solubility is influenced by the electrolyte concentration [26]. It's product a salting-out or salting-in effect, depending on the nature of the ion type. The experimental results also show that the water addition from a mass fraction of 0.22 to 0.29 decreases sucrose solubility in the presence of the studied impurities (Figure 1). The same effect has been observed by different researchers, concluding that the effect of decreasing the water concentration reduces the rate of the hydrolysis reaction of sucrose.









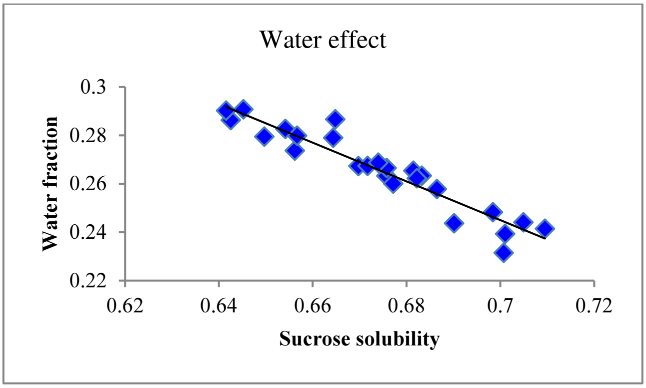


Figure 1. Experimental results of pseudocomponent effect on sucrose solubility, in complex system

Using Pareto chart, the influence of each variable and their interactions on the solubility of sucrose was evaluated statistically (Figure 2). This figure shows that the cumulative effects of three variables contribute 90% of the total effects of sucrose solubility. This is only a main effect of Xw, Xash and Xis. We did not notice any interaction effect between these different variables. The same results were obtained by ANOVA test. Lower P-values are obtained for Xw, Xash, Xis, Xaa and Xac, indicating the main effect significant, but not their squares and interactions. The effect of mass fraction of amino acid and carboxylic acid are relatively moderate. The sucrose solubility can be correlated linearly to mass fraction of water, invert sugar and ash with correlation coefficient of 98.6% (Figure 3), MAE of 0.27% and maximum error of 0.65%. The residual distribution as a function of the predicted values of the sucrose solubility does not show any trend, it is random as can be seen in figure 4.

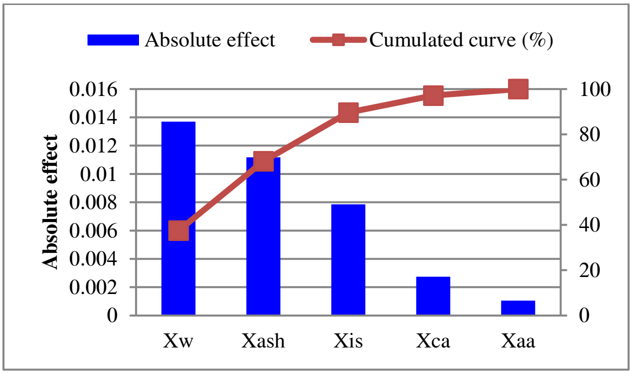


Figure 2. Pareto chart showing the main variables effects on sucrose solubility

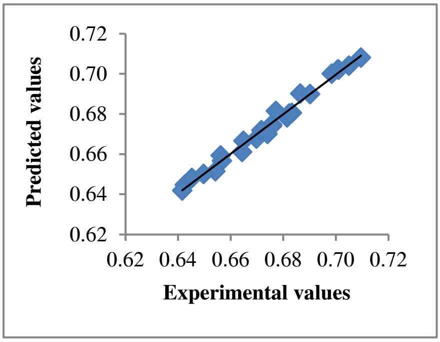


Figure 3. Comparison of experimental and predicted sucrose solubility

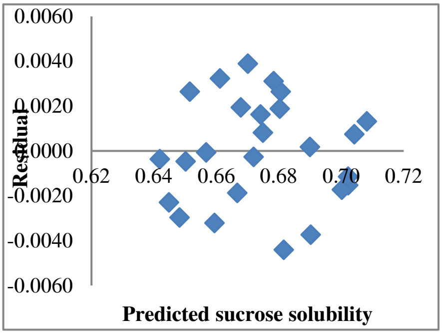


Figure 4. Residual variation in function of solubility predicted value

4.2. Prediction results

New interaction parameters for some group pairs were determined from Experimental VLE data of tree systems: sugar-ash-water, sugar-amino acid-ash-water and sugar-carboxylic acid-ash-water reported by Abderafi and Bounahmidi [20] and from activity coefficient data of binary system: NaOH-H_2O system. For thi calculation, the group parameters of volume, R_k and of the surface area, Q_k, [27], and the interaction parameters available in literature are used. The identification of interaction parameters between groups were determined using the Nelder-Mead method [28]; it consists to minimise the objective function given by:

$$F = \sum_i \sum_j \left(\mathrm{Ln}\,\gamma_{i(\exp)} - Ln\,\gamma_{iUPDH} \right)_j^2 \qquad (14)$$

The values obtained of interaction parameters pairs are shown in table 1, with their estimated error and the values of F. The error of fitted interaction parameters is not exceed 9%.

Table 1. Fitted interaction parameters a_{ij} (K) and a_{ji}

Groups		Interaction parameters		Objective function	Error
i	j	a_{ij}	a_{ji}	F	(%)
CHO	K^+	461.25	-358.7	$1.5\ 10^{-2}$	8.56
CHO	Cl^-	-410	317.75	$1.5\ 10^{-2}$	8.56
COOH	K^+	389.5	-476.63	$1.2\ 10^{-2}$	5.41
COOH	Cl^-	-430.5	3075	$1.2\ 10^{-2}$	5.41
$CHNH_2$	K^+	635.5	-574	$9.8\ 10^{-2}$	5.01
$CHNH_2$	Cl^-	-487.9	594.5	$9.8\ 10^{-2}$	5.01
Na+	OH^-	410.8	-421.37	$3.6\ 10^{-2}$	6.58
H_2O	OH^-	264.41	503.93	$3.6\ 10^{-2}$	6.58
CHO	Na^+	492	-522.75	$1.5\ 10^{-2}$	8.56
CHO	OH-	-471.5	512.5	$1.5\ 10^{-2}$	8.56
COOH	Na^+	615	-563.75	$1.2\ 10^{-2}$	5.41
COOH	OH^-	533	-666.25	$1.2\ 10^{-2}$	5.41
$CHNH_2$	Na^+	625.25	-522.75	$9.8\ 10^{-2}$	5.01
$CHNH_2$	OH^-	-768.75	717.5	$9.8\ 10^{-2}$	5.01
OH^-	H_2O	503.93	-264.41	$3.6\ 10^{-2}$	6.58

Equation (4) and UPDH model (equation 5) were used to calculate SLE data for the studied system at 70°C. The comparison between calculate solubility values and experimental data is represented in figure 5. As shown in this figure, the different points follow the diagonal with a correlation coefficient greater than 50%. The distribution of errors is random between 0 and 7% (Figure 6). These results show that the UPDH method is reliable for the system studied with MAE and ME equal to 3.13 % and 7.20 %, respectively. The large deviation from comparison of the sucrose solubility prediction in aqueous solution with the experimental data has been attributed principally to the kinetic process than to a thermodynamic melting, causing loss of crystal structure in sugars. Hover, the UPDH method gives very satisfactory results.

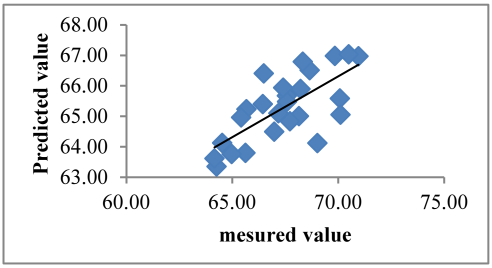


Figure .5: Comparison of experimental and predicted sucrose solubility

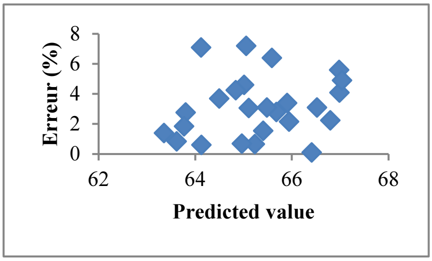


Figure. 6: Error Variation in function of solubility predicted value

5. Conclusion

In the present work, the UPDH model for sucrose solubility prediction in sugar juice was tested, using the pseudocomponent approach. Sugar juice was considered as aqueous solutions of sucrose, invert sugar, amino acid, ash and carboxylic acid. In the first time, the sucrose solubility in a complex system was determined experimentally, for temperature and purity range encountered in industrial sugar process. The experimental results were obtained with a relative error not exceeding 1%. Following statistical analysis, it was found that all the impurities had a significant influence on the solubility of sucrose. Under the operating conditions used; only the salting out effect was observed. This result would be due to the predominant effect of reducing sugars and ash over that of other impurities. Then, the sucrose solubility of the system studied was predicted using the UPDH model. By comparing the predicted values with those obtained experimentally, it can be concluded that this model makes it possible to estimate the sucrose solubility with a very satisfactory error.

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