academicJournals

Vol. 12(12), pp. 1401-1407, 20 March, 2013
DOI: 10.5897/AJB12.1936
ISSN 1684–5315© 2013 Academic Journals
http://www.academicjournals.org/AJB

African Journal of Biotechnology

Full Length Research Paper

Effect of the fructose and glucose concentration on the rheological behavior of high fructose syrups

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Accepted 7 March, 2013

The objective of this work was to study the effect of fructose and glucose content on the rheological behavior of syrups. Initially, high fructose syrup from the fructans present in leaves, bases and head of *Agave tequilana* Weber blue was obtained. Then, its contents of moisture, ash, fructose, glucose and direct and total reducing sugars were determined. Finally, the physicochemical properties of the syrups were evaluated and compared with a high commercial fructose corn syrup (Frudex 55) and other fructose, glucose and sucrose syrups. All of them had the same temperature and concentration of total solids. All syrups behaved as Newtonian fluids and had no statistically significant differences (p <0.05) in their density, water activity and in direct and total reducing sugars. The viscosity and surface tension of syrup depended on its fructose and glucose content. Also, greater fructose content produces syrups with lower viscosity and lower surface tension.

Key words: High fructose syrups, viscosity, rheological behavior, Newtonian fluids.

INTRODUCTION

Nowadays, high fructose syrup (HFS) has displaced sucrose and glucose and has become the sweetener mostly demanded by the pharmaceuticals, food and beverage industries due to its functional and technological advantages (Kovalenko et al., 2010; Montañez et al., 2011b). In the industries, HFS is obtained commercially from corn starch mainly through a process that utilizes various enzymes to hydrolyze starch into glucose and its subsequent conversion to fructose by the glucose isomerase enzyme immobilized in columns on packed matrices (Palazzi and Converti, 2001). The products obtained are syrup, with 42% fructose and the rest are mainly glucose. To obtain syrups with higher fructose content, it is necessary to carry out various stages of separation, conversion, clarification and concentration, within which diverse unitary operations like pumping are performed (Hernandez et al., 2008; Jing et al., 2011).

As a result of the chemical and structural changes which the substrate undergoes from the beginning of the

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Abbreviations: HPLC, High pressure liquid chromatographic; **ADP,** average degree of polymerization; **HFS,** high fructose syrup; **HFCS,** high fructose corn syrup. **TRS,** total reducing sugars; **DRS,** direct reducing sugars; **G,** glucose; **F,** fructose; **DB,** dry basis; a_w, water activity; η, viscosity; τ, shear stress; γ, shear rate; **ºBrix,** soluble solids %; **K,** consistency index; **n,** flow behavior index.

process and to obtain the HFS with concentration and ratio of desired fructose/glucose, in each of the stages of the elaboration process of these syrups, the rheological properties of the fluid change too. In order to achieve an efficient and economic operation, and thus optimize the process, it is necessary to know how fructose andglucose content affects the rheological behavior of the syrups (Corzo and Sanchez, 2008). Knowledge of the rheological behavior of food in general and of high fructose syrups in particular, is of utmost importance for design in process engineering, quality control, as well as in sensory evaluation and determination of food structure (Rao and Ananthesuaram, 1982; Cancela et al., 2005; Dak et al., 2008).

In process engineering, knowledge of the rheological behavior of a fluid is necessary for calculating pumping requirements, establishing the dimensions of pipes and valves, carrying out mixtures and also for calculating basic operations related to heat transfer, mass and movement amount (Corzo and Sanchez, 2008). In quality control, knowledge of the rheological behavior of a fluid is used for quality control of raw materials, as well as intermediary products during manufacturing and of course, of the final products. In sensory evaluation, knowledge of rheological behavior of a fluid helps to determine the preferred quality by the consumer through the correlations between rheological measurements and sensory evaluations (Tabilo and Barbosa, 2005).

Also knowledge of the rheological behavior of fluids helps to elucidate the structure or composition of foods and to analyze the structural changes that occur during its process (Zuritz et al., 2005). Fluid foods exhibit rheological behavior that ranges from simple Newtonian to non-Newtonian fluids (Ibanoglu and Ibanoglu, 1998; Vélez and Barbosa, 1998; Nindo et al., 2005) and the latter can be time dependent or not (Osorio, 2001). Many foods behave as a combination of elastic and viscous materials, and the rheological parameters which characterize them are: viscosity (□), for Newtonian fluids, and consistency index (K) and flow behavior index (n), for non-Newtonian fluids (Rao and Ananthesuaram, 1982).

The objective of this research project was to study the effect of fructose and glucose content on the rheological behavior of high fructose syrups obtained from different botanical sources such as *Agave tequilana* Weber blue fructans and maize starch.

MATERIALS AND METHODS

Ten eight years old *A. tequilana* Weber blue plants were grown in the municipality of Atotonilco el Alto, Jalisco, Mexico. Frudex 55®: high fructose corn syrup (HFCS) with 70% solids, of which 55% is fructose and 45%, glucose (Arancia, Mexico). Mixture of exo-inulinase (EC 3.2.1.80) and endo-inulinase (EC 3.2.1.7) from *Aspergillus niger* in 5:1 ratio, with specific activity of 955 units/mg solid (Megazyme, Ireland) was used. One unit of specific activity was defined as the required amount of enzyme to liberate one micromole of fructose per minute under determination standard conditions. Sucrose, α-D-(+)-glucose and D(-)-fructose, with 99% minimum purity (Sigma, USA). All other reagents were of analytical grade.

Elaboration of high fructose syrups from fructans present in the leaves and head of the *A. tequilana* Weber blue

Initially, the flours were obtained from each of the botanical fractions of the agave plant (tips of leaves, bases and head), which were then used to extract the fructans contained in the same (Montañez et al., 2011a). Later, these extracts were used for the production of high fructose syrups by enzymatic way, with 70% concentration of total solids; they were packaged, labeled and stored dry in the dark and at room temperature for later analysis (Montañez et al., 2011b). By dissolving in distilled and deionized water, syrups of fructose, glucose and sucrose of 70% total solids were also prepared.

Determination of sugars by HPLC

The determination of sugars by high pressure liquid chromatographic (HPLC) was based on the method of Verzele and Van Damme (1986). A liquid chromatograph LDC model 3200 (Variant, USA), equipped with a refractive index detector model 350 (Variant, USA) and a LDC Analytical pump (Variant, USA) was used. A column SPHEREX 5NH2 with 250 mm length and 4.6 mm diameter was used. The mobile phase consisted of an acetonitrile-water solution in 85:15 ratios.The working conditions were: flow rate, 0.4 mL/min; temperature, 35°C and the standards of fructose, glucose and sucrose were injected at a concentration of 2 mg/mL.

Chemical characterization of syrups

The moisture and ash content was determined according to methods of 925.10 and 923.03 (AOAC, 1997), respectively. Direct reducing sugars (ARD) and total reducing sugars (ART) were determined by the method of Miller (1959). The content of fructose and glucose was determined with specific biosensors (Montañez et al., 2006; 2011). All determinations were done in triplicate.

Rheological behavior and viscosity of syrups

The rheological behavior of the syrups was evaluated at a temperature of 25°C, using Rotovisco model Haake RV2 viscometer (Germany) with sensor MV1 at a shear rate of 1000 s^{-1}. In this way, the type of fluid and viscosity (η) of syrups were determined, applying the model of Newton's law of power ($\tau = \eta \gamma$), which relates the shear strength (τ) and shear rate (γ), through the viscosity (η) of the fluid (Rao and Anatheswaran, 1982).

Physicochemical characterization of syrups

The physicochemical parameters evaluated were: density, total solids (ºBrix), water activity (a_w), surface tension, color and viscosity. Density was determined at a temperature of 25°C with a pycnometer Brixco of 25 mL. The total solids (°Brix) were determined at a temperature of 25°C with an ABBE Refractometer (American Optical Co., USA). Water activity was determined at a temperature of 25°C with a Thermoconstanter model RTD33 (Novasina, Switzerland). Surface tension was determined at a temperature of 25°C with a surface Tensiometer model 2141 (Analyte, Australia). Color was determined using a Hunter Lab spectrophotometer with an illuminant D50 and D65 of 10° of the

Table 1. Chemical characterization of syrups.

Parameter	Agave syrup			Corn syrup (Frudex 55®)
	Leave	Base	Head	
Humidity (%)	30.0 ± 0.12[a]	30.0 ± 0.15[a]	30.0 ± 0.01[a]	30.0 ± 0.01[a]
Ash ($\%_{DB}$)	0.31 ± 0.05[a]	0.29 ± 0.05[a]	0.27 ± 0.03[b]	0.05 ± 0.01[c]
DRS ($\%_{DS}$)	99.05 ± 1.25[a]	99.15 ± 1.15[a]	99.55 ± 1.25[a]	99.80 ± 1.30[a]
TRS ($\%_{DB}$)	99.18 ± 1.50[a]	99.30 ± 1.35[a]	99.60 ± 1.10[a]	99.87 ± 1.40[a]
Fructose ($\%_{DB}$)	81.22 ± 0.43[a]	84.54 ± 0.55[b]	87.92 ± 0.28[c]	53.12 ± 1.05[d]
Glucose ($\%_{DB}$)	17.92 ± 0.58[a]	15.20 ± 0.45[b]	11.64 ± 0.36[c]	46.86 ± 1.18[d]

Different letters between columns of the same line indicate statistically significant differences ($p < 0.05$).

observer. It used the CIE-Lab system which involves the parameters, L, a* and b*. Parameter L refers to the brightness of the sample, that is, the degree at which the sample is able to reflect light. Positive values of the parameter a* indicated colorations toward the brownish tone, while negative values indicated colorations toward the green tone. Positive values of parameter b* indicated yellow colorations and negative values of this parameter indicated blue colorations.

Statistical analysis

The results correspond to the average value of three determinations ± standard deviation of the series. Data analysis was performed using SPSS version 12.0 for Windows®. An analysis of one-way variance was conducted and test of Tukey's multiple comparison was done to determine statistical significance ($p < 0.05$) of the physicochemical properties of the different syrups studied. All of them had the same concentration of total solids and temperature.

RESULTS AND DISCUSSION

There was no statistically significant difference ($p<0.05$) in moisture content in the syrup, because they were all prepared with the same total solids content (Table 1). As a consequence of adding NaOH 1 M to 4.25 pH of the agave syrup, for adjustment, its ash content was slightly higher than that that contained corn syrup "Frudex 55®". Industrially, these syrups are passed through ion exchange resins, to minimize their salt content, and thus prevent them from interfering with the organoleptic properties of the syrups, or foods prepared with them (Zhang et al., 2004).

The chromatograms of the different agave syrups obtained (Figure 1) show that such syrups are constituted by the monosaccharides, fructose and glucose, indicating that the hydrolysis of fructan was complete; and therefore its DRS and TRS contents were equal. Moreover, since each fructan molecule contains only one glucose residue and the rest are fructose (Lopez et al., 2003), the content of fructose in the syrups depends on the ADP of the fructans contained in the different agave fractions; if fructans have higher ADP, the content of fructose in the syrup will also be higher. The opposite occurs with the glucose content in the syrup, which decreases when the ADP of the starting fructans increases. The fructose content in the different agave syrups obtained increases in the following order: leaves<bases<head. This is because in this order, the content and the average degree of polymerization of inulin-type fructans present in each of the fractions of agave plant (Montañez et al., 2011a) increase. The fructose content in all agave syrups obtained was much higher than that containing comercial corn syrup "Frudex 55 ®".The content of fructose and glucose in the agave syrups was similar to that containing the syrup obtained in fructans from Jerusalem artichoke (*Helianthus tuberosus*) (Manzoni and Cavazzoni, 1992; Wenling et al., 1999), the main botanical source of proposal for the industrial production of HFS (Jing et al., 2011).

Rheological behavior of syrups

All syrups behave as Newtonian fluids, because the plot of shear strength (τ) vs. shear rate (γ) generates a straight line passing through the origin (Figure 2), and the viscosity coefficient (η) of the syrups is constant throughout the range of shear forces applied (Osorio, 2001).

Another way to determine the flow behavior of a given fluid is to represent graphically the coefficient of viscosity (η) of the cutting speed or shear rate (γ); if this representation generates a straight line of slope equal to zero, this means that the coefficient of viscosity (η) of the fluid does not change and therefore, the liquid in question behaves like a Newtonian fluid type (Rao and Anatheswaran, 1982; Osorio, 2001). Over the whole range of applied shear rate, viscosity of the syrup is almost constant and is zero slope, which means that the viscosity is independent of the applied shear rate (Figure 3), and confirms that the syrups present a rheological behavior for Newtonian fluids. This graphical representation, in addition to viewing the rheological behavior of syrups, is also useful in determining the value of its viscosity (intercept).

Both the density and water activity were virtually identical in all syrups (Table 2), because both are intrinsic properties that are directly related to the moisture content and total solids in the syrup; and since these are equal in

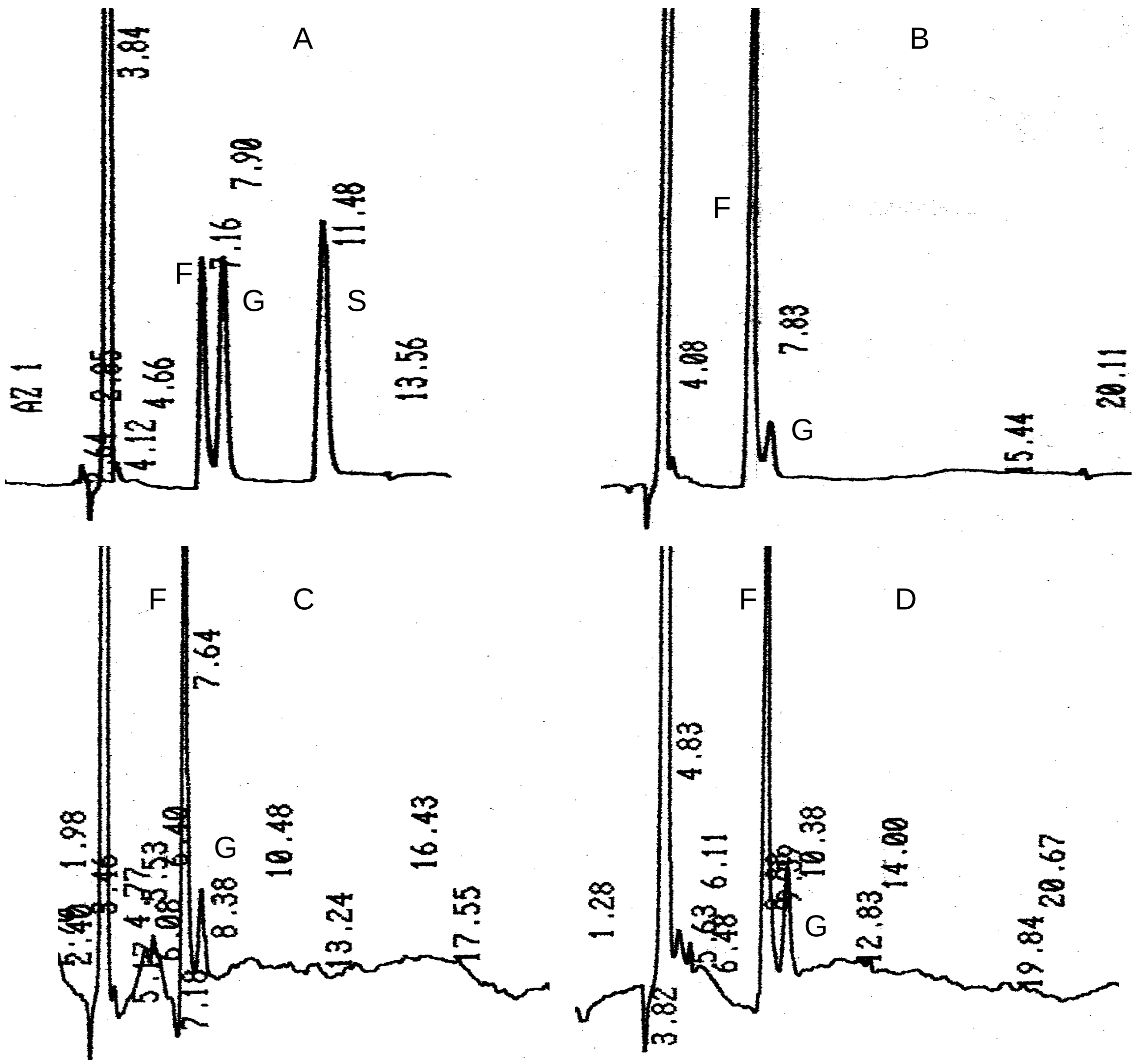

Figure 1. HPLC spectra of: A, Standards of fructose (F), glucose (G) and sucrose (S); B, Agave head syrup; C, Agave bases syrup; D, Agave leaves syrup.

all of them (humidity = 30%, total solids = 70°Brix), so are their density and water activity, meaning that these properties are independent of the composition of syrups (Badui, 1997). The relatively low value of a_w and high concentration of sugars are very convenient for handling and storing the syrup. This is because they prevent the growth of microorganisms therein, without resorting to cooling processes, cooling or addition of antimicrobial agents for its preservation, ensuring the physicochemical and organoleptic properties of the syrups (Hernández et al., 2008).

Meanwhile, the surface tension of syrups depends on the composition thereof and in all cases it was less than the surface tension of pure water (72.75 dynes/cm) (Badui, 1997). Fructose causes a greater decrease in surface tension of the water, followed by sucrose and finally glucose. This is because intermolecular interactions between molecules of glucose are stronger than between molecules of sucrose, and in turn molecules of fructose; therefore, fructose is the most soluble of these sugars (Charley, 1990). In other words, a higher concentration of fructose causes a greater lowering of the surface tension of pure water. This results in a lower surface tension of the syrup, as seen in the values of the surface tension of corn syrups (Frudex 55®), leaves, bases and the head of agave, in which the concentration of fructose in the same increases and the surface tension of the syrups also decreases in that order.

Syrups prepared with pure sugars such as fructose, glucose or sucrose are colorless; meanwhile, the corn syrup Frudex 55® and the syrup obtained from the agave head appear to be clear yellow while the syrups obtained from the bases and agave leaves are orange. The syrup obtained from the agave leaf develops a greater coloring due to higher content of chlorophyll in this portion of the plant, which is easily oxidized and gives a brown color to

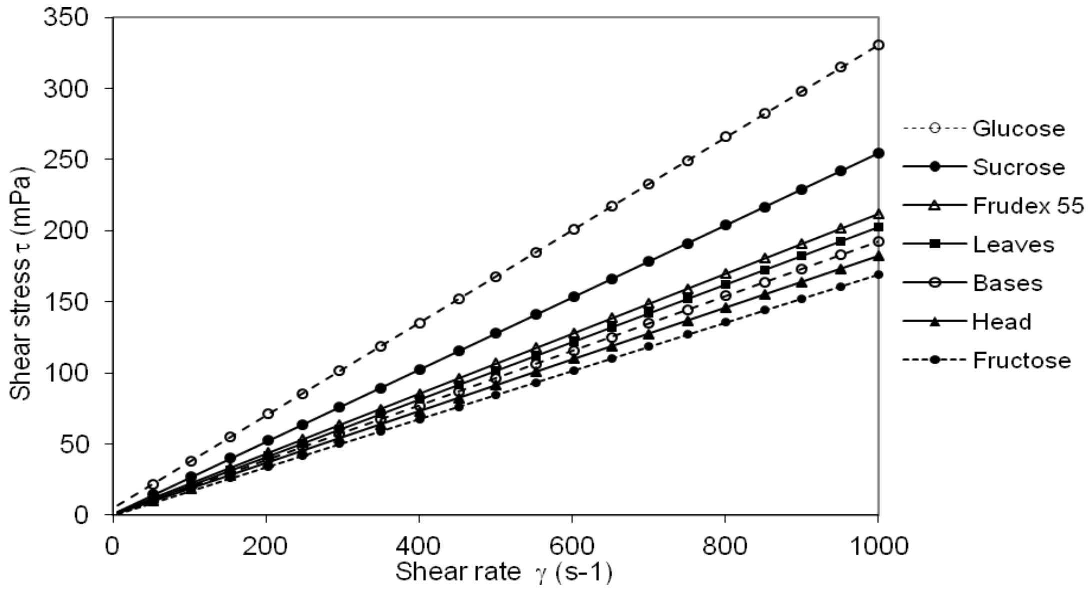

Figure 2. Shear stress vs. shear rate of the different syrups at 70°Brix and 25°C.

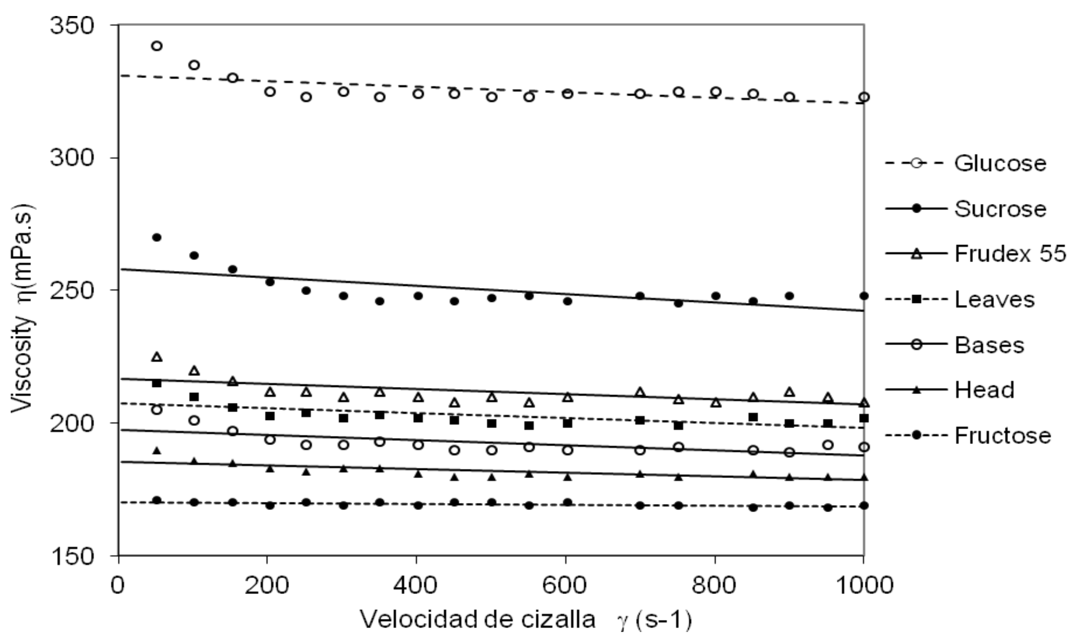

Figure 3. Viscosity vs. shear rate of the different syrups at 70°Brix and 25°C.

the syrup during its elaboration.

Agave extracts compounds derived from Maillard reactions such as furans, pyrans, aldehydes and nitrogen and sulfur compounds such as 5-hydroxymethyl-furfural, methyl-2-furoate and 2.3-dihydroxy-3.5-dihydro-6-methyl-4-pyranone (Mancilla and Lopez, 2002) were identified. Likewise, Kim and Lee (2009) reported the formation of melanoidins as a result of Maillard reactions which occur during the heating of syrups. All these compounds give color to the syrup, which requires its refinement through columns of activated carbon to remove this coloration (Hernandez et al., 2008).

Fructose syrups have a lower viscosity than sucrose syrups, leading to the lower viscosity of glucose syrups (Table 2). For this reason, at the same concentration of total solids, viscosity of the syrup depends on the ratio of fructose and glucose in the same; a higher content of fructose reduces the viscosity of the syrup, while a higher content of glucose increases viscosity of the syrup, as seen in the viscosity values of the different syrups studied

Table 2. Physicochemical characterization of syrups.

Syrup	Density (g/mL)	Water activity (a_w)	Surface tensión (dinas/cm)	Color (a*/b*)	Viscosity (mPa-s)
Glucose	1.51 ± 0.003[a]	0.70±0.002[a]	55 ± 0.6[a]	Colorless	326 ± 4.9[a]
Sucrose	1.50 ± 0.005[a]	0.702±0.001[a]	46 ± 0.4[b]	Colorless	250 ± 4.5[b]
Frudex 55®	1.50 ± 0.005[a]	0.702±0.001[a]	51 ± 0.4[c]	0.01	212 ± 1.5[c]
Leaves	1.50 ± 0.002[a]	0.701±0.002[a]	48 ± 0.5[b]	0.18	203 ± 1.5[d]
Bases	1.50 ± 0.003[a]	0.699±0.002[a]	47 ± 0.5[b]	0.11	193 ± 1.3[e]
Head	1.49 ± 0.003[ab]	0.700±0.002[a]	45 ± 0.6[bd]	0.09	182 ± 1.2[f]
Fructose	1.48 ± 0.003[b]	0.700±0.001[a]	43 ± 0.5[d]	Colorless	169 ± 0.8[g]

Different letters between lines of the same column indicate statistically significant difference ($p < 0.05$).

(Table 2), which decrease in the following order: Frudex 55®>syrup leaves>syrup base>pineapple syrup. In this same order, the content of fructose in the syrup increases.

These results agree with those previously reported by Doty and Vanniken (1975) and more recently by Hernandez et al. (2008), who found that at equal dry matter content and same temperature, the viscosity of the syrup depended on the type of sugar, which was lower in fructose syrups, invert sugar syrup, sucrose syrup and finally by the glucose syrups, whose viscosity was much higher than that of others.

The main factors affecting the viscosity of the solutions are: the nature of the continuous and the dispersed phases, particle-particle interactions and particle-solvent, concentration, shape, particle size and temperature (Osorio, 2001).

The solubility of fructose in water was much higher than that exhibited by the sucrose, and this in turn was greater than the solubility of glucose (Charley, 1990).The lower solubility of glucose was due to a greater degree of particle-particle interactions between its molecules, resulting in an increased viscosity of the solutions; while the higher solubility of fructose is attributed to increased particle-solvent interaction, resulting in a lower viscosity of syrups. In other words, the higher viscosity of glucose syrups with respect to the fructose syrups is attributed to an increased association of glucose molecules in the bulk solution.

An association occurs through hydrogen bonding between them that leads to an effect of highly branched polymer that increases the resistance of the syrup to flow freely; and therefore, increases the viscosity of the system (Osorio, 2001).

Conclusion

The density and the water activity of syrup are independent properties of its fructose and glucose contents; these parameters are equal in all syrups, since they were all prepared with equal content of moisture and total solids. At the same total solids content and temperature, viscosity and surface tension of syrup depends on its fructose and glucose contents; both properties decrease with increasing content of fructose in the syrup or, both properties increase with increasing content of glucose syrups.

Under working conditions established, regardless of their source of origin and content of fructose and glucose, all studied syrups behaved as Newtonian fluids, since the coefficient of viscosity (η) of the fluid was independent on the speed cutting or shear rate (γ).

ACKNOWLEDGEMENTS

The authors thank SIP-IPN, EDI-IPN and COFAA-IPN for the financial support given to carry out this research.

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