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PHYSICOCHEMICAL AND RHEOLOGICAL CHARACTERIZATION OF GOAT WHEY

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ABSTRACT

The whey is a product with high nutritional value, but it is too wasted. In this sense, this study aims to evaluate the physicochemical and rheological properties of goat whey. Content of total protein, lactose, fat, ash, total solids, humidity, pH and density of goat whey were determined. The whey rheological behavior was evaluated in different conditions of temperature and share rate. Two rheological models were used to fit the behavior of the goat whey and the activation energy was determined by Arrhenius equation. The characterization of goat whey showed satisfactory results. The increase in temperature resulted in a decrease of shear stress and apparent viscosity of goat whey, while the reverse was observed for strain rate increased. Ostwald-de-Waelle model presented a satisfactory arrangement with R^2 above 0.99 for all temperatures studied. The goat whey presented behavior index larger than 1, indicating that this is a dilatant fluid.

Contribution/Originality: This study contributes in the existing literature about physicochemical properties of dairy products, focusing in the physicochemical and rheological properties of goat whey. Up to now, nothing was discussed about that. The study also documents that the goat whey is a dilatant fluid with small effect of temperature on viscosity.

1. INTRODUCTION

Whey corresponds to the aqueous portion of milk, which is separated from clot during cheese-production or casein (Kosikowski, 1979). Milk serum, also known as whey, cheese whey or lacto whey, is a byproduct of dairy industry, which for many years was treated only as waste and lost from food industry (Pagno *et al.*, 2009). Although, it is a byproduct of dairy industry, goat whey has significant importance when considering its nutritional composition and production volume. Approximately, 85% to 90% of milk volume used in cheese-production results in serum (Kosikowski, 1979) thereby to manufacture 1 kg of cheese, 10 liters of milk are used, and approximately 9 liters of serum are produced.

The raising goat in Brazil has increased significantly, mainly because it is an alternative for livestock development in the Northeast region. The Brazilian herd of goats corresponds to 8.78 million heads, the Northeast region has the highest effective of this herd, around 8.02 million heads, which corresponds to 91.4% of Brazilian herd of goats (IBGE, 2013). According to *Food and Agriculture Organization* (FAO), goat's milk production in Brazil, in 2012, was 150 thousand tons (FAO, 2012). According to the Brazilian Association of Cheese Industries, in 2013, 1,032 million tons of cheese were produced in Brazil (ABIQ, 2013) which equates to approximately 9.3 million tons of whey. However, few sectors have made use of this nutrients source, leading to a huge waste of a noble food, which may be utilized in diverse food products preparation.

On average, 55% of milk nutrients are retained in the serum, including soluble proteins, lactose, minerals, and vitamins (Kosikowski, 1979). Whey proteins have important nutritional and functional properties, an excellent amino acid profile, and high biological value.

Among the main biological functions of whey protein, it is highlighted its anti-carcinogenic action, anti-inflammatory, anti-microbial, retinol transport, and passive immunity (Poppi *et al.*, 2010) muscles and bones development, cell repair, power supply, and regulation of important metabolic processes (Antunes, 2003) as well as immune modulatory activity, antiviral, and cardiovascular system protection, these proteins present highly digestible, and are rapidly absorbed by the body (Sgarbieri, 2004).

Among different types of whey processing, water separation, proteins recovery, demineralization, and lactose production, are detached. Some whey constituents give special characteristics to food, as water absorption, formation and stability of foam and emulsion, gel setting, viscosity and solubility, and may be utilized as fresh, pasteurized or unpasteurized, condensed and dry, being a great ingredient in new food products development (Mizubuti, 1994). Goat whey is a nutrients source of excellent quality, and should be better utilized, since it is often discarded. This nutrient source has low cost, and may be utilized in a wide variety of food products.

The rheological properties of foods are essential in the evaluation of the ingredients functionality in new products development, as well as in final or intermediate quality control, in shelf life, and in food texture evaluation by sensory data correlation. Thus, rheological behavior evaluation of food products is an analytical tool utilized to better understand the structural organization of food, as well as being important in engineering processes calculations involving multiple equipment, as agitators, extruders, heat exchangers, pumps, homogenizing and pipelines (Steffe, 1996).

The knowledge of food rheological properties also helps in projects and provision of stored, samples stability (Steffe, 1996) since the weather influence on rheological, mechanical and food sense behavior (Silva, 2013). Processing is another important factor that may change a food rheological behavior, due to phases of mixing, heating, cooling, homogenization, aeration, fermentation, crystallization, changing consistency, composition and viscosity modification (Lewis, 1993; Bhattacharya, 1997).

Fluids are classified according to its rheological behavior in Newtonian and non-Newtonian, between those non-Newtonian are thixotropic and rheopectic, which are time dependent; and dilatants, pseudoplastic, and Bingham plastic, which are time independent.

Studies have been performed in order to determine the rheological behavior of milk and its derivatives. Vélez-Ruiz and Barbosa-Cánovas (1998) assessed bovine milk behavior by varying solids contents (12.6% to 48.6%), temperature (5 °C to 25 °C) and storage time (1 to 4 weeks). Milk with solids content up to 22.3% behaved as Newtonian fluid; at concentrations of 24.9% and 30.5% of solid; Ostwald-de-Waelle model worked better for experimental data; as for 42.4 and 48.6 % of solid concentrations, rheological parameters were satisfactorily represented by Herschel-Bulkley model. Silva *et al.* (2012) assessed the rheological behavior of natural yoghurt, reporting that its viscosity decreases with increasing of strain rate, indicating that the yogurt is a non-Newtonian fluid with pseudoplastic behavior. Steffens *et al.* (2005) studied the rheology of prato-cheese, reporting a visco-elastic behavior; he also found that cheeses submitted to a larger stress showed a higher strain and a lower elastic recovery.

By physicochemical and rheological characterization, it is possible to obtain a product quality indicator and its processing. Although goat whey has a high nutritional value, little information is found about its properties and up to now no study has been conducted on the rheological properties of this product. In this sense, this study aimed to evaluate the quality of goat whey, through physicochemical analysis, in terms of the amount of total protein, lactose, fat, ash, total solids, humidity, and values of pH and density, as well as to evaluate the rheological behavior in function of temperature and strain rate.

2. MATERIALS AND METHODS

Goat whey was provided by Association of Small Ranchers of Angicos Backwoods (APASA - Associação dos Pequenos Agropecuaristas do Sertão de Angicos), located in Angicos, RN, Brazil.

2.1. Physicochemical Analysis of Goat Whey

The goat whey was characterized by contents of total protein, lactose, fat, ash, total solids, humidity, pH and density, according to methodologies described below.

- Total protein, lactose, fat and total solids. This determination was performed in DairySpec FT (Bentley Instruments, DairySpec FT manual, USA).

- **Ash.** This was determined by Lanara Method (1981) and was calculated in accordance with Equation I.

$$\%Ash = \frac{100 \times m'}{m} \quad \text{(I)}$$

where: *m* is sample mass, and *m'* is ash mass.

- **Humidity.** Humidity was determined by gravimetric method (AOAC, 1995) and was calculated in accordance with Equation II:

$$\%Humidity = \frac{100 \times m''}{m} \quad \text{(II)}$$

where: *m* is sample mass, and *m''* is dry sample mass.

- **pH.** Whey pH was determined by a pHmeter of Digimed, MD-22model, manufactured in Brazil.

- **Density.** This property was determined by a digital automatic densimeter (Anton Paar, DMA 4500, London, UK), measurements were performed varying the temperature from 25 °C to 50 °C.

2.2. Rheological Behavior of Goat Whey

Rheological measurements for goat whey were determined by a rheometer (Thermo Scientific, Haake Mars, Germany). The analyzes were performed for strain rate from 0 to 1000 s^{-1} for 60 seconds, varying the temperature from 25 °C to 50 °C. Models arrangements to experimental data was performed by using mathematical models of Newton and Ostwald-de-Waelle, through software Origin 7.0. Newton and Ostwald-de-Waelle models are represented by Equations III and IV, respectively.

$$\tau = \mu \gamma \quad \text{(III)}$$

$$\tau = k \gamma^{n} \quad \text{(IV)}$$

where: τ is shear stress (Pa), μ is viscosity (Pa.s), γ is strain rate (s^{-1}), k is consistency index (Pa.s^n), and n is fluid behavior index.

In Otswald-de-Waelle model, the flow behavior index (n) indicates the removal of a Newtonian fluid, in which, for *n* higher than 1 the fluid is considered a dilatant fluid, and for *n* lower than 1, the fluid is characterized as pseudoplastic fluid. Consistency index (k) indicates fluid resistance level to flow.

Temperature effect on rheological profile was also evaluated, being possible to determine the activation energy by using Arrhenius equation (Equation V), in which fluid apparent viscosity present an exponential variation with temperature.

$$\eta_a = A\exp\left(\frac{E_a}{RT}\right) \quad \text{(V)}$$

where: η_a is the apparent viscosity (Pa.s), A is an empirical constant (Pa.s), E_a is the activation energy (kJ/gmol), R is the gas constant (0.008314 kJ/gmol.K), T is the absolute temperature (K).

3. RESULTS AND DISCUSSION

3.1. Physicochemical Analysis of Goat Whey

Table 1 shows the physicochemical properties of goat whey.

Table-1. Physicochemical properties of goat whey.

Physicochemical Properties	This work	Literature	Reference
Protein (% m/m)	0.80 ± 0.31	0.84 to 1.30	(Silveira *et al.*, 2013; Tashima *et al.*, 2013)
Lactose (% m/m)	4.42 ± 0.01	4.51	Tashima *et al.* (2013)
Fat (%)	2.07 ± 0.08	1.41	Tashima *et al.* (2013)
Ash (%)	0.57 ± 0.01	0.6	Tashima *et al.* (2013)
Total Solids (% m/m)	7.62 ± 0.01	7.01	Oliveira (2009)
Humidity (%)	91.81 ± 0.05	92.4	Tashima *et al.* (2013)
pH a 25 °C	6.15 ± 0.01	6.08	Oliveira (2009)
Density at 25 °C (g/cm³)	1.021 ± 0.000	1.027	Oliveira (2009)

Values obtained in physicochemical characterization of goat whey were consistent with those found in literature. In this work, the protein content in goat whey was 0.80% (m/m); similar to obtained by Silveira *et al.* (2013) 0.84%, and lower than reported by Tashima *et al.* (2013) who analyzed the composition of three different whey types (sheep, cow and goat) and obtained a protein content of 0.58%, 0.85% and 1.3% (m/m), respectively, showing a higher protein value for goat whey in comparison with the others. The protein content in the goat whey may vary according to region, besides other factors that go from feed of animal to the processing of cheese, this explain the difference observed in the content of proteins obtained in this work and obtained by Tashima *et al.* (2013). Whey proteins show high nutritional value, and a functional profile not found in other protein (Pagno *et al.*, 2009). Regarding amino acids profile, whey proteins have 42.7% of all essential amino acids, almost all above recommendation, and 6% of tryptophan, higher values when compared to other protein sources. In addition, it is a calcium source with advantages relative to milk, due to its low content of lactose and fat (Haraguchi *et al.*, 2006; Pagno *et al.*, 2009).

Lactose, or milk sugar, is the component present in larger quantities in the whey, which is chemically a reducing sugar compounding most of the total whey solids (Vitti, 1981). Lactose percentage resulted in 4.42% (m/m), a value close to that reported by Oliveira (2009) which was 4.51% (m/m). Lactose is a disaccharide divided in glucose and galactose. This carbohydrate typical from milk is widely used in diverse dairy products manufacturing, as ingredient in childish formula; it is also widely used in pharmaceutical industry as excipient of drugs. In its natural form, it is used as a substrate for fermentation by microorganisms, as in beverages such as fermented dairy (Oliveira, 2006). Lactose has an important role by aiding the calcium absorption and the bone tissue (Vieira and Neves, 1989) besides favoring synthesis of vitamins and reducing fat accumulation in the body (Alais, 1985).

Fat is the milk component with more variation, influenced by factors as standardization of milk, processing, and cheese production (Teixeira and Fonseca, 2008). Fat content of 2.07% (m/m) was higher than those obtained by Tashima *et al.* (2013) which was 1.41 %. Goat whey fat may be utilized in butter, cream, whipped cream and ice cream production, and in cheese production.

Regarding to ash, the goat whey showed 0.57%, which is a result very close to 0.6% reported by Tashima *et al.* (2013). Whey is considered a good source of minerals, consisting of calcium, phosphorus, sodium, potassium, magnesium, and zinc (Baldasso, 2008).

Whey total solids are basically proteins, lactose, minerals salt and fat. Total solids presented 7.62% (m/m), close to 7.01% (m/m) obtained by Oliveira (2009).

Goat whey humidity was 91.8%, value consistent with Tashima *et al.* (2013) who reported 92.4%.

The whey utilized in this study showed pH of 6.15. According to TRIWQ (2013) it is considered sweet if the whey pH ranging from 6.0 to 6.8, and it is obtained when the milk coagulation is mainly performed by enzymatic action. When the pH of whey is less than 6.0, it is considered acid whey, in this case the coagulation of milk is produced mainly by acidification. Therefore, the whey used in this work is type sweet. Goat whey pH obtained by Oliveira (2009) was 6.08, very close to the value found in this study.

Whey density depends on fat level and non-fat solid, because fat has a lower density, and non-fat solids show density larger than water. According to Technical Regulation of Identity and Milk Quality (2002) at 15 °C the milk density may vary in the range from 1.028 to 1.034 g/cm³, density below the established minimum by the Regulation provides water addition indication, and density above of this value indicates creaming, or some corrective product adding. Therefore, this information is very important to identify the quality of final product and check if the milk was adulterated. On the other hand, the Technical Regulation of Identity and Whey Quality (2013) doesn't establish the density of whey. There aren't report about whey adulteration, since it's a by-product of the dairy industry hardly used. In this study, goat whey density was 1.021 g/cm³, value consistent with Oliveira (2009) with 1.027 g/cm³ to goat whey density.

Whey composition may vary according to diverse factors, as milk characteristics, type of cheese produced, heat treatments both used in whey and milk, use of additives, use of ferments, clot rupture time, whey acidity (Oliveira, 2009). Besides production techniques in cheese manufacturing, whey composition depends on seasonal variations, producing species and their food (Rodrigues, 2001). Even with these factors, it is observed that goat whey showed physicochemical properties, consistent with those found in literature.

Figure 1 shows goat whey density variation with increasing temperature.

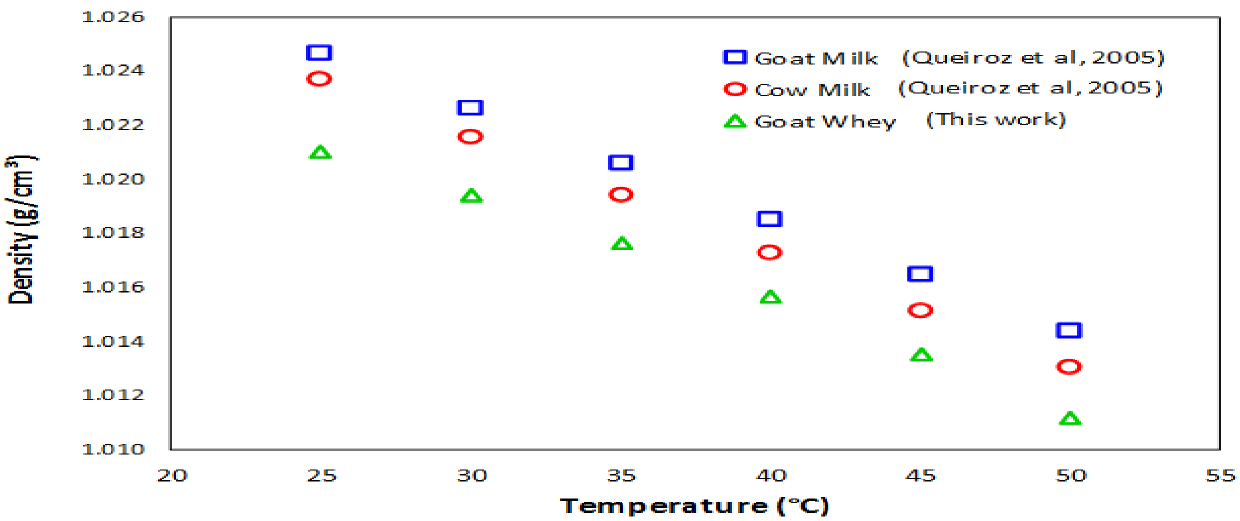

Figure-1. Effect of temperature in the goat whey density.

Figure 1 shows that temperature increasing causes a decrease in density, as described in equation $\rho\ (g/cm^3) = -0.0004T\ (°C) + 1.1389$. Experimental data showed good linear arrangements with R^2 equal to 0.9952. Queiroz *et al.* (2005) assessed thermo-physical properties of goat and cow milk, and observed a decrease in density with temperature in both types of milk, experimental data is satisfactorily adjusted to a linear model described by equations $\rho\ (g/cm^3) = -0.000411T\ (°C) + 1.03499$ with $R^2 = 0.9767$ and $\rho\ (g/cm^3) = -0.00042T\ (°C) + 1.03441$ with $R^2 = 0.9217$ for goat and cow milk, respectively. Plotting these equations in Figure 1, it is observed that at all temperatures measured, the goat whey showed lower density than the goat and cow milk, which is consistent since whey has more fat than the milk (Baldasso, 2008). Since fat solid have lower density than the non-fat solids, the goat whey density will be lower than the milk density. In this sense, the content of fat and non-fat solids is a relevant factor in density evaluation of these products and should be considered in the carried out processes and studies with whey goat.

3.2. Rheological Behavior of Goat Whey

Goat whey rheological behavior by temperature effect and strain rate are shown in Figure 2 and 3.

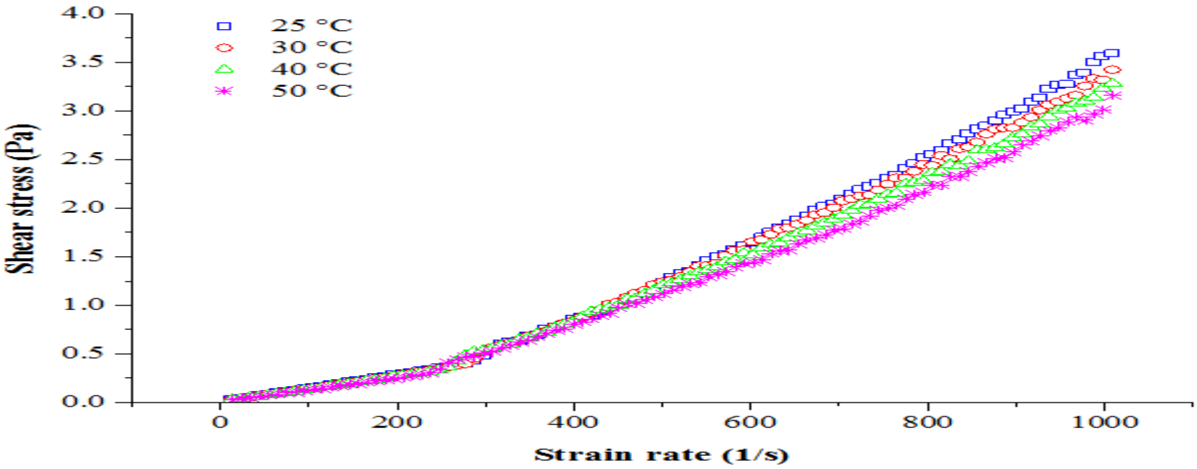

Figure-2. Influence of temperature and strain rate on shear stress of the goat whey.

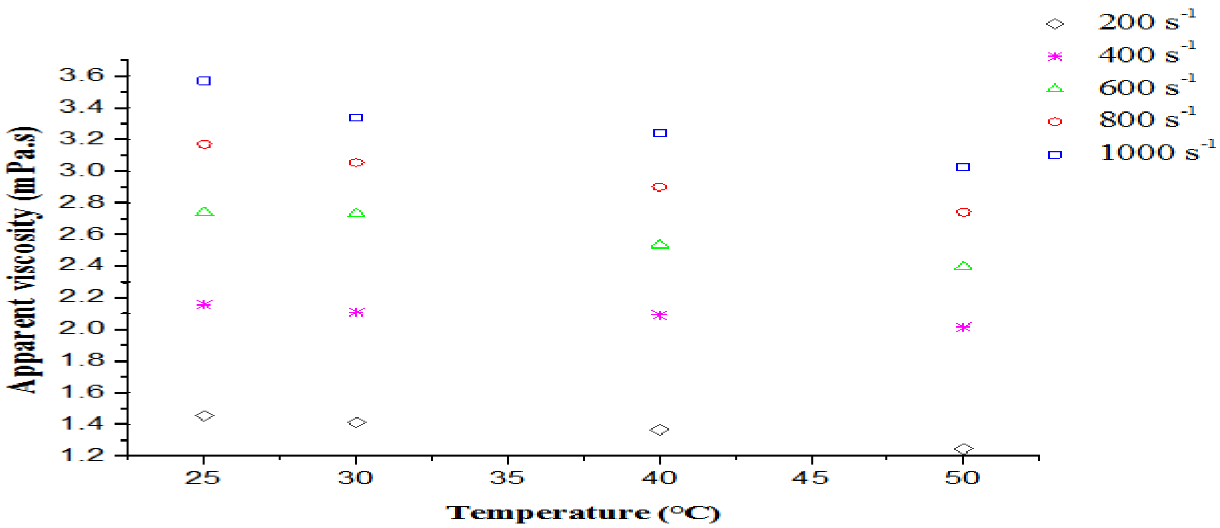

Figure-3. Influence of temperature and strain rate on the apparent viscosity of the goat whey.

Figures 2 and 3 show that temperature increasing resulted in a decrease of shear stress and apparent viscosity in goat whey. According to Pelegrine (1999) the increase in temperature causes a decrease in viscosity, increasing particles movement in suspension.

It also observed that strain rate increase caused an increase in shear stress and viscosity, respectively, indicating goat whey characterization as a dilatant fluid. According to Chhabra and Richardson (2008) the fluid is determined as dilatant when there is an increase of apparent viscosity with strain rate increasing. The friction increases due to structure rupture, and the amount of liquid is not considered sufficient for lubrication. In this sense, fluid behavior may be explained assuming it is composed of dense particles and packed, with small interstitial spaces filled with liquid.

The fitting of experimental data for goat whey was performed by using mathematical models of Newton and Ostwald-de-Waelle. Table 2 shows rheological parameters obtained to each model studied, using goat whey, and varying the temperature.

Table-2. Rheological parameters of goat whey using Newton and Ostwald-de-Waelle models.

Model	Parameters	Temperature (°C)			
		25	**30**	**40**	**50**
Newton	μ	3.04	2.94	2.81	2.65
	R^2	0.9348	0.9440	0.9473	0.9463
Ostwald-de-Waelle	k	0.09	0.12	0.13	0.12
	n	1.5377	1.4789	1.4643	1.4720
	R^2	0.9991	0.9987	0.9993	0.9994

The mathematical model that best fit the experimental data was Ostwald-de-Waelle model, presenting high level of reliability with R^2 above 0.99 for all temperatures studied. Table 2 shows that the flow behavior index (n) is higher than 1 for all temperatures, confirming that goat whey behaves as a dilatant fluid. This same model was used by Silva (2014) to fit the experimental data and evaluates the rheological behavior of buffalo whey; in this study was observed a decrease of apparent viscosity with temperature increasing, characterizing buffalo whey as a pseudoplastic fluid.

Table 3 presents Arrhenius Equation parameters obtained for goat whey in strain rates from 100 to 1000 s^{-1}.

Table-3. Arrhenius Equation parameters for goat whey in different values of strain rates.

Arrhenius Parameters		Strain rate (s^{-1})				
	100	**200**	**600**	**800**	**900**	**1000**
Ea (kJ/gmol)	4.9756	4.7151	4.5756	4.5670	4.4151	4.7968
A (Pa.s)	1.6044	1.5241	0.8273	0.6920	0.5869	0.6772
R^2	0.9941	0.9439	0.9683	0.9974	0.9704	0.9460

Activation energy obtained from Arrhenius Equation for goat whey, except for rate of 1000s^{-1}, decreased with increase of strain rate and showed values of 4.4151 to 4.9756 (kJ/gmol), in all studied conditions it was obtained satisfactory adjust with R^2 ranging between 0.9439 and 0.9974.

Activation energy obtained by Vélez-Ruiz and Barbosa-Cánovas (1998) for cow's milk increased with concentration and storage time, and ranged from 10.12 to 49.37 kJ/gmol. It is known that the higher the activation energy, the more sensitive is the fluid viscosity to temperature changes; therefore, milk viscosity is more sensitive to temperature than whey.

4. CONCLUSION

Goat whey is a product with high added value; its nutritional quality has made this a promising product. In this study, it was possible to verify that goat whey showed 0.80% of protein, a very significant content of protein considering the volume of whey produced in cheese manufacturing. Other chemical compounds amount was within the expected profile for this product.

The density of goat whey presented behavior characteristic for this product, showing to be lower than other products as goat and cow milk, this is a result of the greater amount of fat in the goat whey.

In rheological behavior assessing of goat whey, it was observed that the increase in temperature resulted in a decrease of shear stress and apparent viscosity of goat whey, while the reverse was observed for strain rate increased. This behavior characterized the whey as a dilatant fluid, being evidenced by good arrangement of Ostwald-de-Waelle model, with behavior index value higher than 1. Activation energy obtained from Arrhenius

Equation, varied between 4.4151 and 4.9756 (kJ/gmol), indicating small effect of temperature on viscosity, when comparing this product to other milk products.

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