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Full Length Research Paper

Physical characterisation of some honey samples from North-Central Nigeria

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Some physicochemical properties (water content, sugar content, viscosity, pH and conductivity) were determined for honey samples from North-Central Nigeria to evaluate their global behaviour and comparison with other honey samples. The water content and sugar content varied within the range of (18.22 - 36.82%) and (63.82 - 80.25%) respectively. The pH increased with increase in water content and the conductivities of the samples had correlation with proportion of minor constituents in the honey samples. The relationship among water content (w), temperature (t) and viscosity (η) for different honey samples of may be represented as $\eta = 17.678\times 10^3 \exp(-0.32w - 0.088t)$. The temperature dependence of viscosity was evaluated with Arrhenius model, the activation energy with value of 70.07 kJ/g is fairly unaffected by moisture content.

Key words: Honey, viscosity, characterisation, water content, pH, sugar content.

INTRODUCTION

Natural honey is one of most widely sought products because of its unique nutritional and medicinal properties. These properties had been attributed to the influence of separate groups of substances it contains (Aduku and Olukosi, 2000). Natural honey is a sweet aliment produced by honey bees from sugary solution of nectar of flowers as their source of food in time of scarcity or during harsh whether condition. Natural honey is composed of mainly of sugars (glucose and fructose) and water. Other constituents include: other types of sugars, proteins, minerals, phytochemicals (such as organic acids, vitamins, and enzymes etc), as well as other substances that may serve as sources of dietary antioxidants. Each of these minor constituents was known to have distinctive nutriational or medicinal properties and the unique blend account for the varied different applications of natural honeys.

Artificial honeys can be produced from carbohydrate sources that have glucose-fructose composition that are within a close range with that of natural honey. These artificial honeys often have similar taste and physical appearance as natural honeys, but they lack the medicinal and nutritional properties of natural honeys because of the absence of the minor constituents that are present in natural honeys. It has also been observed that the composition of minor constituents of natural honeys varies with location/nectar sources and different climatic conditions. This informs the need for characterisation of honey samples. Although, the tasks involved for complete analysis of a complex product like honey is not only laborious but also expensive. However, some physicochemical properties of honeys that can be easily determined have been found to be helpful for comparison of natural honey samples from different locations and also serves as important indicators that can help to distinguish natural honey from artificial honey. The physical-chemical properties provide the parameters for characterisation and classification of honeys. They also serve as criteria used for choosing appropriate processing and packaging technique, and technological applications of natural honeys (Bhavesh and Bhattacharya, 2002). Popek (2002) used the physical-chemical parameters to classify seventy three honey samples. Using only three parameters (total ash content, total acidity and dynamic viscosity), a nearly correct classification (98.67%) was achieved.

The procedure for processing and storage method of a given sample of honey depends greatly on its moisture content. This is because it bears a direct relationship to

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the likelihood of undesired fermentation caused by osmophilic yeasts. Honey samples with carbohydrate content < 83% and moisture content > 17.1% are easily prone to fermentation especially when they are kept at temperatures > 11°C. A well processed honey is expected to have a long shelf life and should not ferment.

The viscosity of honey is essential to its processing and it has an important link to its technological applications as a supercooled aqueous sugar solution. Honey of high quality is usually thick and viscous. The viscosity of a honey sample is a function of the composition of its sugars, water and colloid content. If the concentration of water is increased, honey becomes less viscous. Proteins and other colloidal substances increases honey viscosity, but their amount in honey may be insignificant. The percentage of fructose content in honey has also been found to affect is viscosity and rheological properties. Honeys become less viscous with increase in fructose content. The viscosity of various honey samples from different sources around the globe had been reported (Karoui et al., 2007; Graciela et al., 2004; Hayette et al., 2007; Al-Khalifa and Al-Arify, 1999). The chemical properties of honey samples from different locations of southern part of Nigeria had been reported. There is yet a report on the physical characterisation and viscosity study of honey samples from Nigeria. Thus, in this report we present the results of viscosity study and physical parameters of honey samples from north-central Nigeria. The physical parameters used to characterise the honey samples in this investigation are water content, sugar content, density, pH, conductivity and viscosity.

MATERIALS AND METHODS

Honey samples

The honey samples used in this study were obtained from four local markets designated A, B, C and D in Kwara State, North-Central Nigeria. Before being used, they were heated up to 55°C to dissolve any crystals in the samples, and kept in flasks at 30°C to remove air bubbles which could interfere in viscosimetric studies. Four different aqueous solutions [20, 40, 60 and 80 $(^{w}/_{w})$] of each sample of the honey were prepared, and the exact water content was determined by the method of Abu-Jdayil et al., 2002.

Refractive index

Refractive index and Brix grades were determined using an Abbe refractometer. The refractometer was first calibrated with doubled-distilled. Water was circulated into the instrument through a thermostatically controlled bath, and was maintained at ± 0.1°C. The refractive index measurements were taken after the honey attained a constant temperature in the refractometer. This was done in triplicate and average value was taken for the experimental values.

Density

The pycnometry method was used to determine the density of the honey samples and their solutions. The densities of the samples were determined using the expression below:

$$\rho = \frac{[W_2 - W_1] \ (g)}{V \ (cm^3)}$$

Where;
W_1 – mass of the pycnometer when empty
W_2 – mass of the pycnometer when filled with a honey sample
V – volume of the pycnometer

pH

The pH of the honey samples and their solutions were measured using a 211 microprocessor pH meter.

Conductivity

The conductivity of the honey samples and their solutions were determined using Jenway 4510 Conductivity Bridge with a cell constant 1.56 cm. The probe was dipped into the samples and the conductance reading Φ_s, was taken for each of the samples. The conductance Φ_{dw}, of doubled-distilled water used for preparing the aqueous solutions of the honey samples was also measured. The conductivity values κ_s, for each sample was calculated from the relation: $\kappa_s = (\Phi_s - \Phi_{dw}) \, x \, 1.56$.

Viscosity measurements

The capillary viscometric measurements were performed using an Ostwald viscometer. The viscometer was initially calibrated using doubled distilled water and 20% sucrose solution. Calculated amount of distilled water was added to the fresh honey sample to raise percentage water content to 25 - 50 range. The viscosities of the samples were measured for each of the samples at 15, 25, 35, 45 and 60°C.

RESULTS

The result of this work is represented in Table 1, Figures 1 – 5.

DISCUSSION

The refractive index values of aqueous solutions of the honey samples are presented in Table 1. The water content and the Brix (sugar content) of the honey samples and their solutions were calculated from the respective refractive index values. The water content was determined by adopting the expression (Equation 1) developed by Abu-Jdayil and co-workers (Abu-Jdayil et al., 2002).

$$\% \text{ Water} = 608.277 - 395.743 \cdot n_D. \qquad (1)$$

Where; $n._D$ - refractive index

The water and sugar content of the honey samples is represented in Figure 1. It was observed that samples A, B and D have water and sugar content between (18 - 23)%

Table 1. Refractive index, percentage water content and percentage sugar in the honey samples and their solutions.

Honey samples	A			B			C			D		
Sample solution %	n_D	water (%)	Brix grade	n_D	water (%)	Brix grade	n_D	Water (%)	Brix grade	n_D	Water (%)	Brix grade
20	1.355	72.05	32.71	1.357	71.25	33.41	1.355	72.04	32.71	1.356	71.65	33.05
40	1.377	63.34	40.40	1.38	62.15	41.45	1.376	63.73	40.05	1.389	58.59	44.59
60	1.398	55.03	47.74	1.393	57.01	45.99	1.400	54.24	48.44	1.399	54.63	48.09
80	1.442	37.62	63.12	1.423	45.13	56.48	1.421	45.93	55.78	1.421	45.93	55.78
100	1.479	22.97	76.06	1.491	18.22	80.26	1.444	36.82	63.82	1.486	20.20	78.50
		99.03			98.48			100.64			98.70	

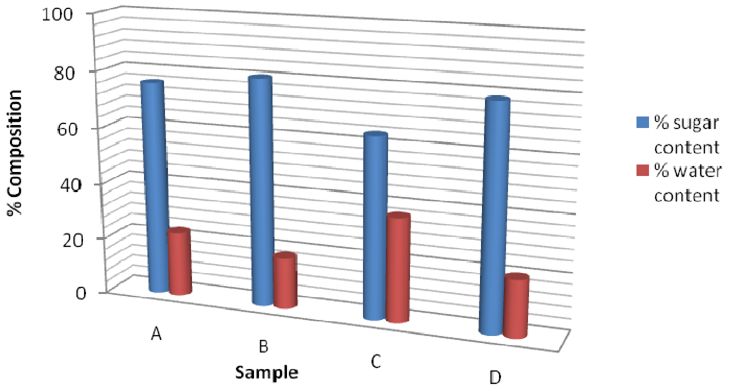

Figure 1. Percentage water and sugar content in the honey samples

and (78 - 80)% respectively. The overall sum of the water and the sugar content constitute (98 - 99)% and the remaining (1 - 2)% could be attributed to non-sugar components of these samples. These values are within the range obtained for samples from southern Nigeria and similar to what is obtained for samples around the globe. The result presented in Figure 1 also revealed that the sugar content of sample C is lower and the water content is higher than that of the other samples. The sum total of the water and sugar content for sample C constitute 100% of the sample. This probably suggests absence of non-sugar (minor) components which accounted for the unique properties that are critical to the applications of natural honeys. It therefore implies that sample C could be an artificial honey from cane sugar.

The percentage water content/activity is a quality parameter which is used to estimate the shelf-life, staling rate and crystallisation rate of honey samples. Honey samples that have high water content are readily prone to high staling and high crystallisation rate. Such honey samples will be rendered non-homogeneous and the quality and market value will be affected. Consequently the percenttage water in honey samples is usually reduced by heat-treatment to a level where the microbial activities is reduced to the minimum so to maintain the homogeneous quality, ensure they have acceptable shelf-life and enhance their market acceptability. On the basis of percenttage water and sugar content, honey samples are usually graded voluntarily using USDA standard (Table 2).

Table 2. Percentage water and total sugar (USDA standard).

Grade	water content %	minimum total sugar %
Good	≤ 18.6	81
Reasonably good	≤ 20	80
Low	> 20	< 75

Thus, based on the results presented in Table 1 and the criteria shown in Table 2, sample B and D in their fresh untreated form can be adjudged to be good and reasonably good grade honey samples respectively, while A and C are of low grade, susceptible to fermentation. Samples A and C needs different level of heat treatment. The quality of honey may be adversely affected by too much heat. The commonly employed heat treatment procedure are: short duration of high temperature heat treatment (subjecting the sample to a temperature of 77°C for two minutes followed by rapid cooling to 54°C); or long duration of low temperature heat treatment (keeping the honey at 60°C for 30 min); or at 77°C for one minute or a straight line gradient between these tempera-

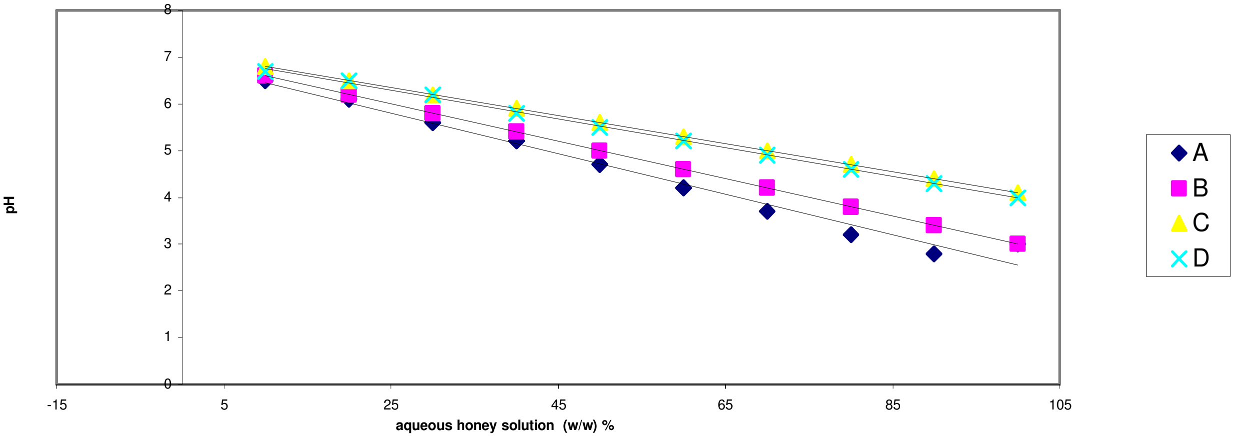

Figure 2. pH versus concentration of aqueous solution of the honey samples

these temperatures. Sample A has about 5% water content above the USDA standard; it requires less amount of heat treatment. Sample C has about 18% water content above the USDA standard for grade A. The short duration high temperature heat treatment procedure is therefore prescribed for sample A, and long duration of low temperature heat treatment procedure is prescribed for sample C.

Honey is a mixture of sugars but contains also among other minor constituents, amylase (enzyme), which can hydrolyse starch to dextrin and/or simple sugars and the optimum pH for honey amylase activity is (5.3 - 5.6). The effect of honey type and concentration on starch gelatinization had been reported by Sopade et al., 2004. The identified trend is that starch viscosity decreases as the pH of the system neared the optimum. The result depicted in Figure 2 shows the pH of aqueous solutions of the honey samples. The pH of the fresh samples without added water content is 3.0, 3.0, 4.1 and 4.0 for A, B, C and D respectively. Comparison of the pH values with % water content of the fresh samples did not reveal any obvious relationship. However, there is correlation between percentage non-sugar constituents and conductivity. The conductivity values of the honeys are 0.0975, 0.0147, 0.00003 and 0.0468 $\Omega^{-1}m^{-1}$ for samples A, B, C and D respectively. The inverse of the gradient of the graphs of pH against concentration (Figure 2) is an indication of the buffering capacity of the honey. This shows a direct relationship to the amount of minor constituent of the honey samples. Sample C, suspected to be artificial honey has the buffering capacity; this is attributable to lack of minor constituents in the sample. Honey from different sources (differing in pH and amylase activity) show a varied effect on starch gelatinization. Between pH 3.0 and 4.0, starch viscosity was unaffected by the honey irrespective of the source. However, above this pH values, starch viscosity decreases as the pH approached the optimum for honey amylase activity (5.3 - 5.6). The viscosity did not increase as the pH moved away from the honey amylase activity optimum. Differences in pH of honey samples and a model sugar mixture had been identified, and their effects on starch gelatinization were attributed to honey amylase activity, composition and concentration of minor organic compounds present.

The viscosity of natural honey samples have been reported to be affected by temperature, moisture content and floral source. But the global behavior of natural honey samples in relation to their viscosity values are usually determined using an empirical multiple regression relation (Equation 2) developed by Junzheng and Changying. In the equation the viscosity of a honey sample is expressed as function of its water content and temperature.

$$\eta = a.e^{(b.w + c.t)} \qquad (2)$$

Where; η is the viscosity; *w* is the water content; *t* is the temperature in Celsius; and *a*, *b* and *c* are adjustable parameters.

The parameters a, b and c provides a means of characterising honey samples. The influence of temperature and water content on the viscosity of the honey samples are presented in Figure 3.

The values obtained for the adjustable parameters for the tested honey samples (17.678 m Pa·s, -0.32%$^{-1}$ and -0.088℃$^{-1}$ respectively). These values are similar to those obtained by Chinese honeys and Galician honeys (Junzheng and Changying, 1998; Gomez-Diaz et al., 2005). It was observed that in all the samples the influence of the variables (temperature and % water content) on the viscosity was high at their low values.

Sugars are used as sweetener/thickener in food products. The extrusion of aqueous solution of a sugar depends on the viscosity which in turn is also a function of the interactions between the components in the system. It can be said that at low % water content the average intermolecular distance between the sugar molecules is small; this enhances strong sugar-sugar hydrogen bonding and also promote entanglement between the oligosaccha-

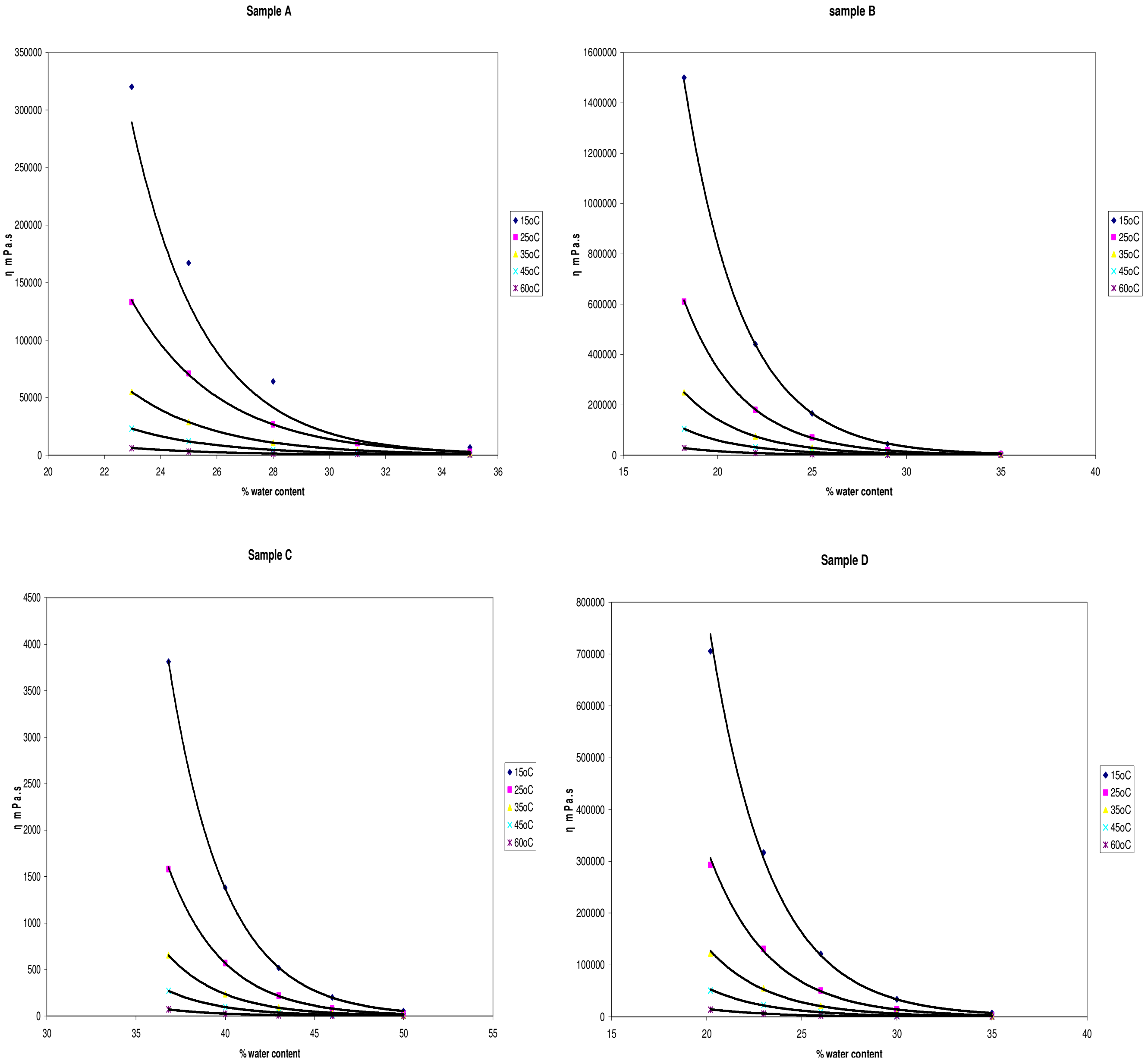

Figure 3. Effect of water content on viscosity at different temperatures.

rides. This accounts for the thickness and high viscosity at this state in the absence of crystallization. The behaviour will tend towards glass-like properties with further reduction in the % water content. Addition of water to honeys in their natural form brings about exchange of the sugar-sugar hydrogen bonding with sugar-water hydrogen bonding (hydration). This being favoured size of water (solvent) molecules compared to that of the sugars thus permitting better interaction and resulting in isolation of the sugar(s) within the solvent medium. This accounts for the drastic decrease of the viscosity of honey even at low values % added water. The influence of temperature can also be attributed to ease of breaking the less efficient sugar-sugar hydrogen bonds, due to steric hindrances which is also enhanced by water molecules. The rheological properties of honey are important qualities that influence the sensory quality of the product and also affect a number of technological operations, such as honey heating, mixing, filtering, hydraulic transport and bottling. It is generally agreed that honey have Newtonian properties. However, there have also been reports indicating a possibility of non-Newtonian behavior of some honeys (Sławomir, 2007). Rheological behaviours of honey can be investigated with models such as Arrhenius, Vogel–Tammann–Fulcher (VTF), Williams–Landel–Ferry (WLF) and Power Law (Brenda et al, 2003; Sopade, 2003; Yoo, 2004; etc). These models are used to assess the temperature sen-

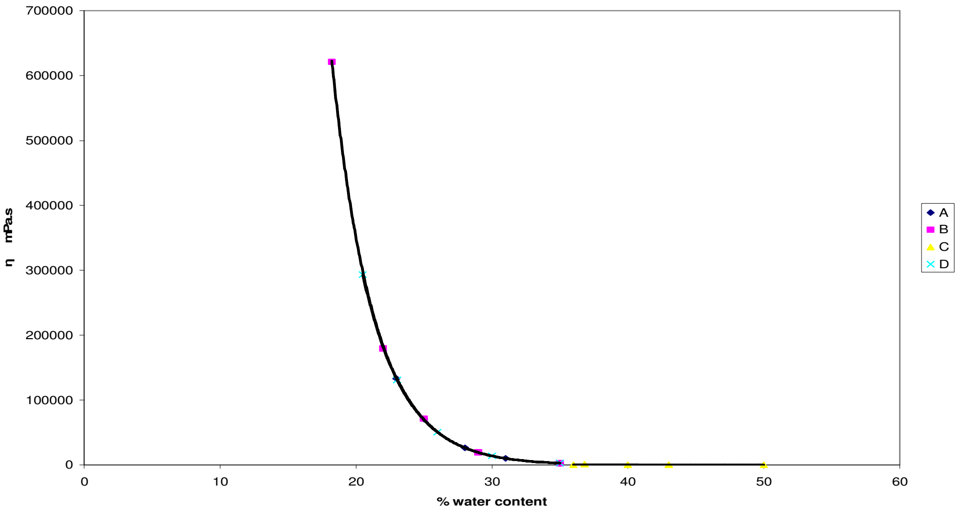

Figure 4. Effect of water content upon the viscosity value for all honeys at 25℃.

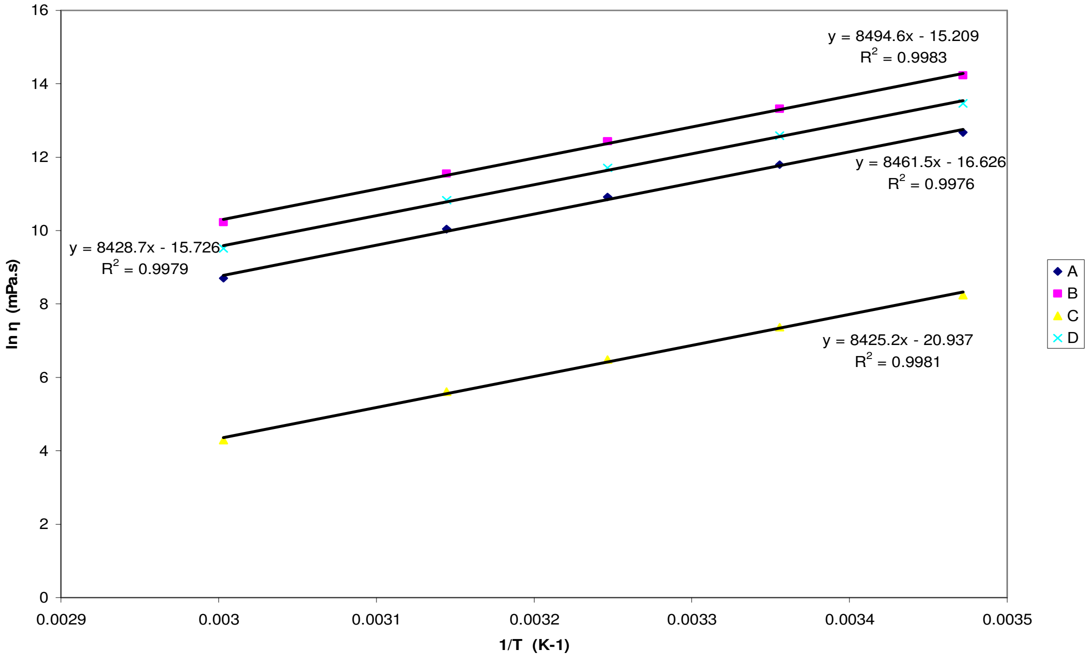

Figure 5. Arrhenius plot for viscosity of the honey samples at different temperature.

sitivity of the honeys viscosities Arrhenius model was employed to evaluate the rheological behaviors of the honey samples. The Arrhenius activation energy obtained was 70.07 kJ/g (Figure 5). The value was fairly unaffected by moisture content, but the values of the Arrhenius constant η_o correlate linearly with the percentage water content of the honey samples.

Conclusion

The physico-chemical properties of four honey samples obtained from four local markets in Kwara state, North-central Nigeria was determined and used to evaluate their global behaviour in comparison with other reported honey sample around the globe. The percentage water content was an important parameter used to access quality of honey samples. It was found that percentage water content could serve as an indicator to detect an artificial honey sample disguised as a natural honey sample. The result of the study revealed that two of the four honey samples meet the quality requirement of the USDA standard for honey samples, while the other two required different level of processing in order to upgrade them to the USDA standard

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