Journal of Experimental Research

Dec. 2015, Vol 3 No 2
www.er-journal.com
Email: editor-in-cheif@er-journal.com

Received: November 2015
Accepted for Publication: December 2015

Refining and Characterisation of Gum Arabic Using Vacuum Filtration Method for Application in Oil and Gas Drilling Fluid Formulation

[1]Bilal S*, [2]Mohammed-Dabo IA, [3]Dewu BBM, [4]Momoh OR and [5]Abubakar S.

[1,2,4& 5] Department of Chemical Engineering, Ahmadu Bello University, Zaria - Nigeria.
[3]Centre for Energy Research and Training, Ahmadu Bello University, Zaria - Nigeria.

***Corresponding author email: bsabiu@abu.edu.ng**

ABSTRACT

This research focused on refining, characterisation and determination of the physicochemical properties of raw and refined Gum Arabic. Vacuum filtration method was used for the refinement and both the raw and refined samples were characterised using Fourier TransformInfrared (FTIR) analysis. Both the raw and refined samples were odourless and dark brown in colour with the pH and viscosity values of 4.7, 63cP and 5.4,124cP respectively. The solubility of the raw and refined Gum Arabic in cold water at 18^{0}C and ethanol were 13.57%w/w, 0.01%w/wand 14.32%w/w,0.04%w/w respectively.The solubility curve indicated **that the solubility of Gum Arabic increases with an increase in temperature.** On the other hand both the raw and refined sampleswere insoluble in acetone and chloroform.The FTIR spectrum give the presence of functional groups such as alkyl halides, alkanes, alkenes, aliphatic amines, aldehydes and phenol that are common to polysaccharides and a clear level of impurities removed from the raw Gum Arabic that enhanced its viscosity for application as a viscosifier in drilling fluid formulation.

Key words: GumArabic, characterisation, FTIR, Viscosity, Acetone and Chloroform

INTRODUCTION

Acacia species exudate is a natural resin that contains arabin; a semi solidified sticky fluid emerging from incision made on bark of *Acacia* trees (Nuhu*et al.,* 2009). *Acacia* trees belong to the botanical family of Leguminosae, a predominant species of the group of Mimosaceae. There are more than two hundred species of *acacias*, but only a few of them produce gums. The only species producing gum arabic, as per United Nations Food and Agriculture Organization (FAO) definition, are *Acacia senegal* and *Acacia seyal* which have different properties and are also divided into several varieties (Gaudiose et al., 2012).

A study shows that there are about 17 species producing commercial Acacia gum in Sub-Saharan Africa. Most of these gums are edible and some are used in the food and pharmaceutical industries (FAO, 1996).

Nigeria produces different grades of exudates and is ranked as second largest world producer after Sudan, together they produce about 45,000 tons of gum arabic to the market each year (Partos, 2009). Gary and Ryan (2002) reported that the trees grow more in Borno, Yobe, Sokoto, and Bauchi states of Nigeria. The trees are used as potent weapon in the fight against land desertification and soil degradation in sahelian belt of the country without industrial uses (Nuhu et al., 2009).

Four major commercial grades of gum Arabic are produced in commercial quantities in Nigeria (Osagie, 2002), namely: (1) *Acacia* Senegal (hard gum):-This is known as grade 1 and it is sourced mostly from Yobe and Borno states of Nigeria. (2) *Acacia* Seyal (friable gum):-It is known as grade 2, sourced mostly from Bauchi and Jigawa states in Nigeria. It has a positive optical rotation. (3) Combretum: It is commercially called grade three, has a negative optical rotation and is sourced from the fourteen producer states of Nigeria. (4) Neutral:-It is the fourth variety of gum Arabic. It does not have definite biological name but called neutral grade.

Gum Arabic is a complex mixture of polysaccharides, protein and arabinoglacto protein species. It has been shown to be highly heterogeneous and is found in nature as mixed calcium, magnesium, potassium and sodium salts of a polysaccharic acid or arabic acid

(Omar et al., 2013).

Hirst et al.,(1989) reported that the major Acacia species is used for pharmaceutical, confectionary, food, textile and beverage production among others. Gum Arabic is currently widely used in food and non-food industries where it functions as an emulsifier, stabilizer, thickener, flavouring or coating agent (Wickens et al., 1995). These functions are associated with certain quality requirements fulfilled by gum properties such as absorption, tastelessness, odourlessness, solubility, viscosity and rheological behaviour (Glicksman, 1969).

The presence of impurities in the gum Arabic reduces the quality of the gum, as such; the need to remove these impurities arises in order to improve the gum arabic quality to an acceptable standard (Khan and Abourashed, 2010).

The locally produced Gum Arabic in Nigeria contain some impurities that may affect their viscosities when used as additives for drilling fluid formulation, hence there is need to remove the impurities such as sand, dirt, pieces of bark and leaves and insects among others in order to enhance their properties. This research work is aimed at refining and characterization of Gum Arabic (Acacia gum) using vacuum filtration method.

MATERIALS AND METHODS

The methodology employed in the refining of gum Arabic is explicitly summarized as follows:

Sample Collection

The raw Gum Arabic sample shown in plate 1 was procured from Sabon Gari Zaria Market, Kaduna State. The sample is relatively pure, however impurities such as pieces wood and broken leaves were carefully removed by hand and then air dried until it became sufficiently brittle. Then the raw Gum Arabic was reduced to smaller particle size using jaw crusher and milled to fine powder using ball milling machine. The powdered sample depicted in plate 2 were kept in an air-tight plastic container and stored at room temperature before the analysis.

Plate 1: Raw Gum Arabic sample

Plate 2: Fine powdered Gum Arabic sample

Gum Arabic Solution Preparation

100g of gum Arabic powder was dissolved in 200ml of distilled water and allowed to stand for 24 hours with intermittent stirring to ensure complete dissolution of the gum. The gum mucilage was then strained through a filter cloth into a basin to remove impurities in order to obtain particle-free slurry which was allowed to sediment. Thereafter, the gum supernatant was precipitated from the slurry by addition of 350 ml of 96% ethanol.

Vacuum Filtration Set-up

The Buchner flask was placed on laboratory table where the funnel was inserted into the neck of the flask and the vacuum rubber tube was attached to the side arm of the Buchner flask. The filter paper was then wetted with a small amount of water and placed into the funnel where it stick on the funnel body and the vacuum pump was turned on as depicted in Plate 3..

Plate 3: Gum Arabic Refining Set-up

Plate 4: Purified Gum Arabic sample

Filtration Process

The precipitated Gum Arabic solution prepared, was poured into the Buchner funnel contain filter paper and this was done in such a way that the Buchner funnel was filled to the top (brim) in order to exert enough pressure to push down the solution through the Buchner funnel. The solution that passed through was then collected in a Buchner flask and discarded. The purified residue as depicted in plate 4, was collected and put into basin. Finally, the residue was air dried and milled as refined Gum Arabic ready for characterization.

pH Determination

The 60ml solution of refined Gum Arabic was poured into a beaker and the pH was determined at 24.6^{o}C using 3510pH meter. The same procedure was used to determine the pH of raw gum Arabic solution.

Viscosity Determination

The viscosity of 60ml solution of raw and purified gum Arabic were determined at 24.6^{o}C using a Fann Viscometer (Model 35SA) at 300rpm .

Solubility Deyermination in Various Solvents

The solubility of the raw and refined Gum Arabic sample was determined in various solvent (water, acetone, chloroform and ethanol) and also in water at different temperatures 25^{0}C, 45^{0}C, 65^{0}C, 85^{0}C and 100^{0}C. 1g sample of the gum was added to 50 ml of each of the above mentioned solvents and allowed to stand for 6 hours. 25ml of the clear supernatants was weighed and poured into crucible and put in desiccator until a permanent weight was achieved. The weights of the residue with reference to the original weights of the samples were determined using weighing balance and expressed as the percentage solubility of the gums in the solvents in weight by weight.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectrophotometer (8400S) was used to characterize both the raw and refined samples in order to identify functional groups of the chemical constituents in the samples. The FTIR analysis was carried out at the National Research Institute for Chemical Technology (NARICT), Basawa, Zaria.

RESULTS AND DISCUSSION

The result obtained for the physicochemical properties of the raw and refined Gum Arabic such as colour, odour, taste, pH, Viscosity, Solubility, as well as their characterization using FTIR are presented as follows:

Physicochemical Properties of Gum Arabic

The physicochemical properties of the raw and refined Gum Arabic such as colour, odour, pH, taste and viscosity were carried out and the results are depicted in Table 1. The colour of the raw sample is orange–brown while that of the refined sample is dark-brown. This change in colour may be attributed to the type of solvent used for the purification process. The raw and purified Gum Arabic had a bland taste and are odourless. The pH of raw and purified gum Arabic were 4.7 and 5.4 indicating mild acidity. This is due to the fact that gums are generally macromolecular acids. The acidity of plant gums is obvious since most of them are known to contain salts (Ca, Mg, K, Na, and Fe) of acidic polysaccharides, the acidity of which is due to uronic acids in their structure (Odeku and Fell, 2004). The higher acidity of the raw gum in comparison to the refined Gum was also due to the type of solvent used in their purification.

Table 1: Physicochemical properties of Gum Arabic

Property	Raw Gum Arabic	Refined Gum Arabic
Colour	Orange -brown	Dark -brown
Odour	Odourless	Odourless
Taste	Bland	Bland
pH	4.7	5.4
Viscosity (cP)	63	124

The viscosities of raw and refined Gum Arabic were 63 and 124 cP respectively. The lower viscosity value for the raw gum was attributed to the fact that it has less resistance to flow due to the presences of impurities in the main structure which weaken and break the bond. These impurities affect the viscosity of the gum by preventing the polysaccharide chains in the gum to interact inter molecularly. Different metal ions present in Gum Arabic molecules may also affect its viscosity. The high viscosity of purified sample was attributed to the impurity removal from the raw samples.

Solubility in Various Solvents

The solubility of the raw and refined gum in various solvents such as cold water, chloroform, acetone and ethanol are presented in Table 2. The solubility of the raw and refined sample in cold water at 18^{0}C and ethanol were 13.57%w/w, 0.01%w/w and 14.32%w/w, 0.04%w/w respectively. The solubility of the refined Gum Arabic in both cold water and ethanol are slightly higher than that of the raw Gum Arabic and both samples were insoluble in chloroform and ethanol. The low solubility of the raw gum is due to the presence of impurities in the gum such as high molecular weight molecules and insoluble matters. This conform to the discovery of Sarah et al. (1998) which explained that Gum Arabic is ionic in character because it dissolve in water and partially dissolve in ethanol. It shows high solubility in water (14.32 %w/w) and partial solubility in ethanol (0.04%w/w).

The low solubility of the gum observed in ethanol is due to the fact that it can ionize to produce hydroxyl ion (OH^-) and the value of its dielectric constant is higher than those of acetone and chloroform. Consequently ethanol has some polar character over acetone and chloroform which are characterized by low values of dielectric constant. On the other, the partially solubility of the gum in ethanol may be due to the presence of polar and non-polar ends in ethanol.

Table 1: Solubility (w/w %) of Gum Arabic in different solvents

Solvents	Solubility (%W/W) Raw Gum Arabic	Solubility (%W/W) Refined Gum Arabic
Cold water (18^{o}C)	13.57	14.32
Acetone	0.00	0.00
Chloroform	0.00	0.00
Ethanol	0.01	0.04

Figure 1 shows that the solubility of both the raw and refined samples increase as the temperature values increases from 25 °C to 100 °C. The highest solubility values for the raw and refined samples observed at 100°C are 59.42 %w/w and 87.22%w/w respectively. The solubility of natural gum depends on the ratio of soluble to insoluble matters. Therefore the increase in solubility could be related to a reduction of insoluble matter during refining. The difference in solubility between the raw and refined samples was higher at 65 °C and lower at 25°C. This shows that the effect is more observed at higher temperature.

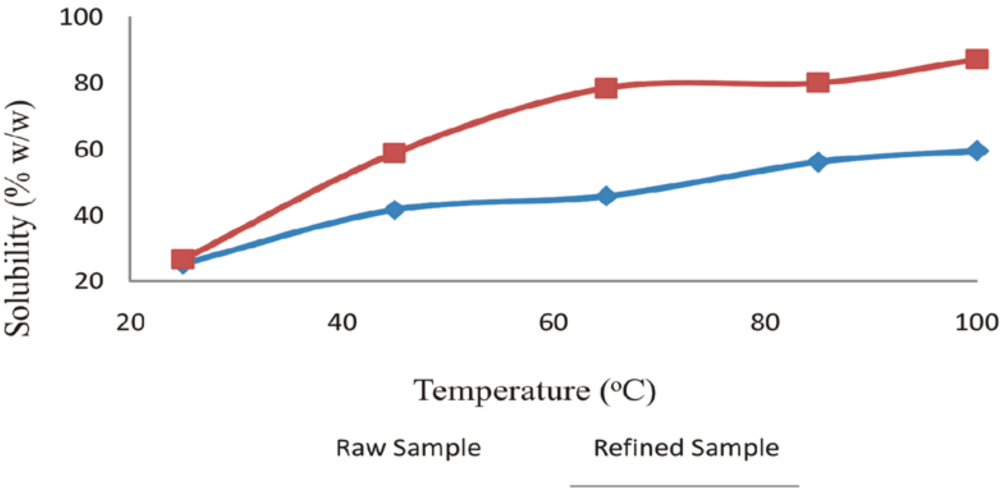

Figure 1: Solubility (% w/w) of Raw/Refined Gum Arabic in Water at various temperatures

The low solubilities of the raw sample at the different temperatures is due to the presence of impurities and inorganic matter. It has been found that the solubility of some ionic solutes tends to vary with temperature due to the change of properties and structure of liquid water. An increase in temperature can increase the degree of solute-solvent interaction resulting in increase in solubility. It is evident that the raw gum Arabic exhibited poor solubility.

FTIR Characterization Result

The FTIR spectra of raw and refined Gum Arabic are shown in Figure 2 and 3. The gum Arabic spectra showed a typical characteristic of absorption band for common polysaccharides. The assignment of prominent peaks for the raw and refined Gum Arabic was given in Tables 3 and 4 respectively.

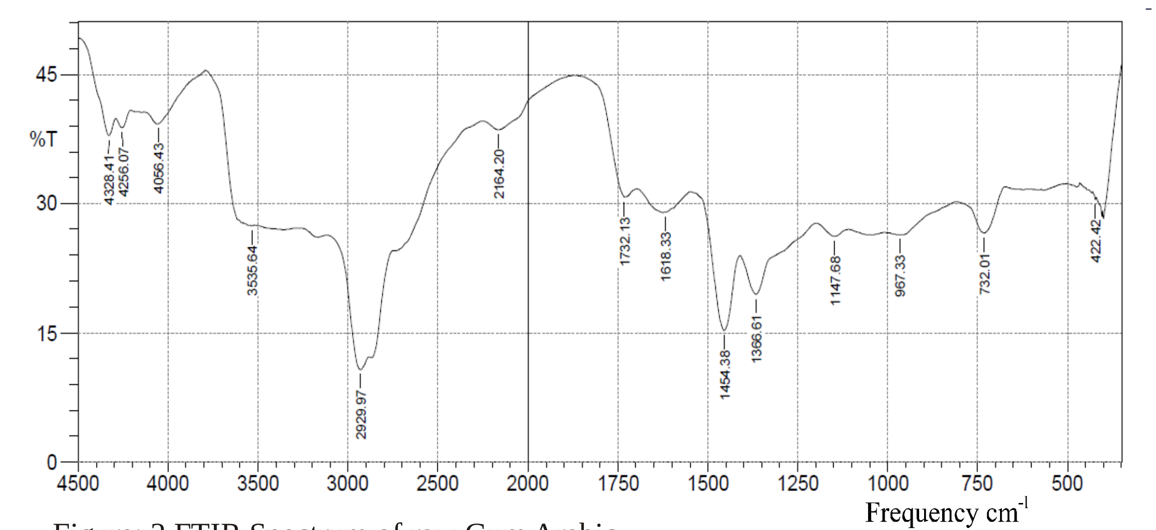

Figure: 2 FTIR Spectrum of raw Gum Arabic

Table 3: FTIR Characteristics of raw Gum Arabic sample

S/No	Frequency, cm^{-1}	Bond	Vibration	Functional group	Intensity
1	422.42	-C-I	Stretch	Alkyl halides	30.382
2	732.01	-C-H	Rock	Alkanes	26.59
3	967.33	=C-H	Bend	Alkenes	26.359
4	1147.68	C-N	Stretch	Aliphatic amines	26.201
5	1366.61	C-H	Rock	Alkanes	19.473
6	1454.38	C-H	Bend	Alkanes	15.239
7	1618.33	N-H	Stretch	1^0 amines	28.978
8	1732.13	C=O	Stretch	Aldehydes	30.751
9	2164.20	- C≡C -	Stretch	Alkynes	38.566
10	2929.97	C-H	Stretch	Alkanes	10.699
11	3535.64	O-H	Stretch	Alcohol or phenol	27.419

The FTIR spectrum of raw Gum Arabic in Figure 2 displayed features, typical of polysaccharides. Thus C-I (alkyl halides) stretch vibration was found at 422.42 cm^{-1}, C-H rocking at 732.01 cm^{-1} due to alkanes. The =C-H bend vibration of alkenes is association with 967.33 cm^{-1}, C-N stretching due to Aliphatic amine at 1147.38 cm^{-1}, C-H rocking and bending vibrations at 1366.61 and 1454.38 cm^{-1} due to Alkanes. Other vibrations included N-H stretching at 1618.33 cm^{-1} due to 1^0 amines, -C≡C- stretching at 2164.2 cm^{-1} due to C-H stretching at 2929.97 cm^{-1} due to alkanes. The frequency number 1732.13 represent C=O stretching functional group of aldehydes, saturated as well as O- H stretching at 3535.64 cm^{-1} due to alcohol or phenol group.

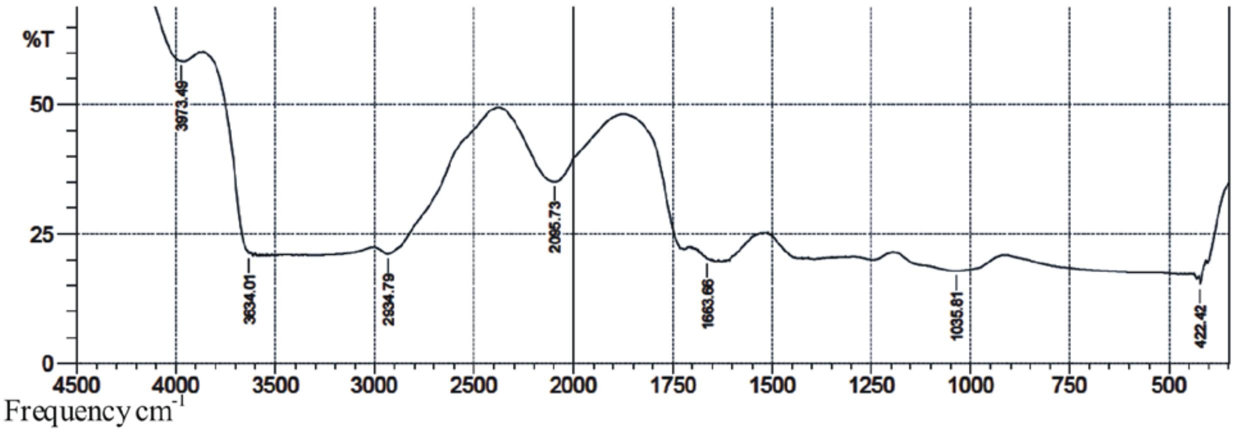

Figure.3 FTIR Analysis of Refined Gum Arabic

Figure 3 presents FTIR spectrum of purifiedgum while Table 4 presents bonds, vibrations, intensities and frequencies of IR adsorption as well as associated functional groups. The spectrum displayed features, typical of polysaccharides. Thus -C-I stretch vibrations were found at 422.42cm^{-1} due to alkyl halides. The peak obtained at 1035.81 cm^{-1} is due to stretching modes of C-N bonds of aliphatic amines and -C=C-stretches due to Alkenes that were prominent at 1663.66 cm^{-1}. Other functional groups identified were -C≡ C- stretching vibration at 2095.73 cm-1 due to Alkynes, C-H stretch due to Alkanes at 2934.79 cm^{-1} The band 3634.01 cm $^{-1}$ denote O-H stretching of Alcohols or phenol. These functional groups are present in material like carbohydrate, starch and some other natural gum (Malik et. al., 2002).

Table 4: FTIR characteristics of purified gum Arabic sample

S/N	Frequency, cm-1	Bond	Vibration	Functional group	Intensity
1	422.42	-C-I	Stretch	Alkyl halides	15.259
2	1035.81	C-N	Stretch	Aliphatic amines	17.822
3	1663.66	-C=C-	Stretch	Alkenes	20.225
4	2095.73	-C≡C-	Stretch	Alkynes	35.1
5	2934.79	C-H	Stretch	Alkanes	21.115
6	3634.01	O-H	Stretch	Alcohols or phenol	21.203

Tables 3 and 4 depicts the functional groups for both the raw and refined gum Arabic. The functional groups include alkylhalides, alkane, alkenes, alphatic amines, 1^{o} amines, aldehydes saturate, alkynes and alcohol/phenol which are essentially found in Gum Arabic (complex polysaccharides). However, there was an observed shift in the frequency of the functional groups for the refined Gum Arabic. It was also observed that C-N stretch (aliphatic amine) at 1147.68 cm^{-1} shifted to 1035.81 cm^{-1}. –C=C- stretch (alkenes) 967.33 shifted to 967.33 cm-l. -C≡ C- stretch (alkynes) at 2164.20 cm^{-1} shifted to 2095.73 cm^{-1}. C-H alkanes stretch shifted from 2929.97 to 2934.79 cm^{-1}. O-H stretch (alcohol/phenol) at 3535.64 cm^{-1} shifted to 3634.01cm^{-1}. These shifts in frequency after adsorption show that some functional groups have been removed from the raw Gum Arabic. Alkanes were found to be the predominant functional groups present in both raw and refined Gum Arabic samples which conforms with the previous studies.

CONCLUSION

The conclusions from the research are as follows:

1. Refining of the Gum Arabic has an effect on the physicochemical properties such as pH, viscosity, colour, and extent of it solubility.
2. The raw Gum Arabicwas light brown which turned to dark brown with pH values of 4.7 and 5.4 respectively. The two samples were odorless with bland taste.
3. The viscosity of the raw and refined Gum Arabic were 63cP and 124 cP respectively, showing that the refined gum is more viscous due to removal of impurities from the raw gum sample.
4. Functional groups identified in the gum were found to be closely related to those of other polysaccharides and strongly reflected its chemical constituents.
5. The variation of solubility with temperature at constant time indicated that the solubilities of the gums in water increased with an increase in temperature. But the raw and refined Gum Arabic were insoluble in acetone and chloroform.

6. The Refined gum Arabic can therefore be used as an additive in drilling fluid as a viscosifier to enhance properties of the fluid, if the impurities are properly removed.

REFERENCES

FAO (United Nations Food and Agriculture Organization), (1996).A Review of production, markets and quality control of gum arabic in Africa. Project No. TCP/RAF/4557, FAO.

Gary H, Ryan R, (2002) Best Practices, Markets, Training. Nigerian Gum Arabic Producer and Small Trader. *A Workshop Workbook*, Lagos.

Mujawamariya G, Burger K, D'Haese M (2012). Quality of Gum Arabic in Senegal: Linking the Laboratory Research to the Field Assessment. *Journal of International Agriculture* 51(4); DLG-Verlag Frankfurt/M.

Glicksman M. (1969).Gum Technology in the Food Industry. *Food science and technology*, 8 Academic Press, New York.

Hirst EL, Jones JK, Smith F, (1989). Plant gums local vegetable oils. Ph.D Metallurgical Engineering and Mucilage, *Advanced Carbohydrate Chemistry*, pp: 243-250.

Ibrahim OB, Osman ME, Hassan EA, (2013). Characterization and simple fractionation of Acacia Senegal. *Journal of Chemica Acta*.11-17.

Malik H, Gupta N, Sarkar A (2002). Anisotropic electrical conduction in gum arabic—A biopolymer. *Material Science Engineering* 20: 215–218.

Nuhu A, Ademoh A, Abdullahi AT (2009).Determination of the Physio-Chemical Properties of Nigerian *Acacia* Species for Foundry Sand Binding Applications. *Research Journal of Applied Sciences, Engineering and Technology* 1(3): 107-111.Maxwell Scientific Organization.

Odeku OA, Fell JT (2004). Evaluation of Khaya gum as directly compressible matrix system for controlled release. *Journal of Pharmacology.* 56: 1365-137.

Osagie C, (2002). Gum Arabic and Diversification of Nigerian Economy. *This day Publishing Co. Limited.* Lagos, Nigeria.

Partos L (2009). Sudan scraps gum arabic monopoly to liberalise market. In: http://www.foodnavigator.com/Financial-Industry/.

Sarah LV, Singer BW, Hictchen SM, Townsend JH (1998).The Development and initial application of a gas chromatographic method for the characterization of gum media. *J. Am. Inst. Conversation* 37:295311

Wickens GE, AG Seif El Din, Guinko S, Ibrahim N (1995).Role of *Acacia* species in the rural economy of dry Africa and the Near East. *FAO Conservation guide* 27, Rome.