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PURIFICATION, CHARACTERIZATION AND MODIFICATION OF GUM ARABIC FOR POSSIBLE USE AS ADDITIVE FOR POLY VINYL CHLORIDE

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ABSTRACT

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Gum Arabic exudate was collected from Acacia senegal trees around Zaria metropolis, purified in 95% ethanol and its physical and chemical modifications carried out. Physical modification of the gum involved plasticization of the gum with glycerol and ethylene glycol. The chemical modification was performed by acid hydrolysis, acetolysis and acetate formation. Appearance of both modifications was observed after three days of drying. Acetic anhydride (AAH), acetolysis (ACT) and ethylene glycol (EGL) modifications became hard and solid, and were ground to powder. Glycerol (GLY) turned very sticky and acid hydrolysis (AHY) turned into a viscous liquid. From characterization of the samples, all modifications were found to be less dense than the pure gum Arabic sample (PGM). AHY sample was found to be more turbid and has the highest conductivity value followed by AAH sample. pH of all the samples were found to be below 7.0, indicating acidic nature of the gums. Melting point measurements showed that all test samples have lower melting point values than the pure gum. The viscosities of aqueous dispersions containing different concentrations of pure gum Arabic and that of the modified gums were determined. When concentration was varied for each gum, the viscosities of the solutions increased with increasing concentration. At each concentration the viscosity of AHY is far below that of the other samples, this could be attributed to the fact that acid hydrolysis of polysaccharides normally breaks polysaccharides down to their monomers, dimers and other oligosaccharides. FTIR spectra of the pure and chemically modified samples were studied. It was found that there were shifts and absorptions at different frequencies, indicating degree of interaction between the gum and the modifying solvents.

INTRODUCTION

Gum arabic is an edible, dried, gummy exudate from the stems and branches of *Acacia senegal* and *Acacia seyal* that is rich in non-viscous

soluble fibre (Williams and Philips, 2000). It is a neutral or slightly acidic salt of a complex polysaccharide containing Ca²⁺, Mg²⁺ and K⁺. Its most distinguishing property among

the natural gums is its extreme solubility in water. The gum is obtained from trees of genus; *Acacia*, subfamily; *Mimosoidene*, family; *leguminosae* (Smolinske, 1992). Gum arabic is the result of an infection, either bacterial or fungal. It is exuded only by unhealthy trees; heat, poor nutrition and drought stimulate its production. The gum is exuded through these wounds in the bark in the form of tears, or drops which rapidly harden due to evaporation (Glicksman and Sand, 1973).

Gum Arabic is a highly heterogeneous material, but could be separated into three major fractions. Most of the gum (88.4% of total) is arabinogalactan with 0.35% protein content and molecular weight of 3.8×10^5 Da. The second fraction (10.4% of total) is an arabinogalactan protein (AGP) containing 11.4% protein and 1.45×10^6 Da molecular weight. The third is a low molecular weight glycoprotein (GP) 1.2% of the total gum, has a protein content of 47.3% and a molecular weight of 2.5×10^5 Da (Randell *et al.*, 1989).

Gum arabic is insoluble in alcohols and most organic solvents. Aqueous solutions are slightly acidic with pH range of 4.5-5.5. Viscosities of gum arabic solutions are relatively low with a viscosity of 200cps having been reported for a 30% solution. Maximum viscosity is attained at pH 6-7, with only a gradual change over the pH range of 5-10. Partial hydrolysis may occur at low pH. The viscosity also increases gradually as the concentration increases from 2-25%, at which point a more marked increase takes place, but it is inversely proportional to temperature. One of the major functional characteristics of gum arabic is its ability to act as an emulsifier for essential oils and flavors. Prolong heating of gum arabic solution causes proteinaceous components to precipitate out of solution, thereby influencing the gum's emulsification properties (Williams, 2000)

Light-scattering data suggests that gum arabic molecules are in the shape of short, stiff spirals with numerous side chains. The length of the coil is 1050Å. Hydrolyzing gum arabic yields L-arabinose, L-rhamnose, D-Galactose and D-glucuronic acid. The glucosidic bonds in gum Arabic vary in liability but preparations appear to have common

structural features. The main structural feature of gum arabic is a back bone of (1→3) – linked D-galactopyranose units joined to the main chain by 1,6 – linkages (Verbeken *et al.*, 2003).

Espinosa-Andrews *et al.*, (2008), studied chitosan-gum Arabic coacervates and quantified the amounts of polysaccharides in soluble phase and insoluble phase (coacervates). They used high pressure liquid chromatography (HPLC) technique to quantify monosaccharides produced after acidic hydrolysis in soluble phase. They also prepared Chitosan–gum Arabic coacervates at different pH and further evaluated micro-structural and rheological characteristics of the coacervates. They found that coacervates were formed due to the interaction of the functional groups of both macromolecules ($-NH_3^+$ and COO^-), using Fourier Transformation Infrared Spectroscopy (FT-IR).

Osman *et al.*, (1993) fractionated GA by hydrophobic interaction chromatography to yield four fractions, all of which had a similar carbohydrate composition, but differed in their concentration of protein, amino acid composition and molecular mass distribution. All four fractions reacted with an array of anti-arabinogalactan protein monoclonal antibodies via anti-carbohydrate epitopes and were precipitated by Yariv's reagent, which indicated that all four fractions were AGPs. It was shown that at least one protein was unique to each fraction.

NMR spectroscopy has been shown to be a powerful analytical method for the unambiguous identification of GA; samples adulterated with other gums are readily detected using this technology (Anderson *et al.*, 1999). As-Assaf *et al.*, (2005) found that analysis of GA using GPC coupled to a multi-angle laser light scattering detector, a refractive index detector and an UV detector set at 214nm provided a useful “fingerprint” for the rapid evaluation of GA for specific applications. The method was able to assess protein content and its distribution in the various gum components.

PVC additives have brought forward a host of peculiar problems that have continually posed challenges to the ingenuity of mankind.

PVC additives in a broad variety of ways, both good and bad, have affected the life of just about every civilized person on the face of the earth. PVC in toys, children and infant products and packaging often contain toxic additives such as phthalates, organotins, lead and cadmium to soften or stabilize the plastic. These additives are not chemically bound to the plastic and can migrate out of the product posing unnecessary toxic hazards to children and consumers (Quevauviller, 1991; Hoch, 2001). In recent years, a growing body of scientific evidence has found that toxic chemicals released by the PVC lifecycle have been found in the bodies of babies, children and adults. Babies are being born pre-polluted with potentially harmful levels of phthalates and dioxin that may possibly cause lifelong health problems (Latini *et al.*, 2003).

This study seeks to investigate and compare the characteristic of gum Arabic and its modifications using standard methods and to provide evidence of the gum modification for possible use as cheap and locally produced additive for PVC.

MATERIALS AND METHOD

MATERIALS USED

The reagents used include: chloroform, ethanol, petroleum ether, concentrated sulphuric acid, acetic acid, acetic anhydride, glycerol and ethylene glycol were obtained from the British Drug House. The equipment used include: Brookfield's Viscometer (Brookfield Engineering Lab, Inc. Middleboro, MA, USA), pH Meter (CRISON micropH 2000, Spain), FTIR-8400S Fourier Transform Infrared Spectrophotometer and GCMS-QP2010 plus Shimadzu Japan.

COLLECTION AND PURIFICATION

The trunk of the tree *Acacia senegal* in Zaria Petropolis was incised and the gum exudates were allowed to dry and then handpicked from the trees. The dried gum was washed and dried in hot air oven at 40°C for 48 hours. The gum was then crushed to break up and then hydrated in double strength chloroform water for five days with intermittent stirring. The mucilage obtained was strained through a clean calico cloth and the gum was precipitated using 95% w/v ethanol. The precipitate gum was filtered,

washed with diethyl ether then dried in hot air oven. The dried gum was pulverized and passed through sieve size No 60 (250µm), (Oluyemisi *et al.*, 2010).

CHARACTERIZATION

pH Determination

The pH of 1% w/v of the pure and modified gums was determined with a microprocessor based pH Meter (Model 1012, Esico, Mumbai, India). The pH meter was set to neutral at room temperature and the electrode was immersed into the solution.

Viscosity Measurements

2% w/v of dry gum and each gum modification were prepared and then diluted to concentrations of 1.81, 1.67, 1.54, 1.42, 1.33, 1.25, 1.17, 1.11, 1.05, and 1.0% w/v. The viscosities of the different concentrations were determined using Brookfield viscometer (Engineering Lab Inc., Middleboro, MA, USA.)

Melting Point Determination

A melting point capillary tube was used. A tiny sample of the gum powder was placed on a piece of weighing paper. A few crystals of the compound were then placed in a thin walled capillary tube 10-15cm long, about 1 mm in inside diameter, and sealed at the other end. The capillary tube, which contains the sample, and a thermometer are then suspended so they can be heated slowly and evenly. The temperature range over which the sample was observed to melt is taken as the melting point.

Salinity, Conductivity and Total Dissolved Solid (TDS)

These were determined using a conductivity meter (Hach Sension5 Conductivity Meter)

Density Measurements

Density analysis of 1% w/v of the test sample was carried out using a density bottle. The weight of the empty bottle was measured (W) and then weight of the bottle with 70ml of the test sample as W₁). The difference was taken to get the actual weight of the liquid, this weight was divided by the volume to obtain the density.

FTIR Analysis

FTIR is commonly used for qualitative identification of various functionalities. The FTIR spectrum of the powdered gum was recorded with a Perkin Elmer RXI spectrophotometer (Connecticut, USA).

The dry powder was mixed with KBr and pressed into pellets. The spectrum was obtained by scanning between 4000 and 500/cm.

GUM MODIFICATION

Both chemical and physical modifications were carried out on the gum Arabic.

Chemical Modification

This was performed by acid hydrolysis, acetolysis, and acetate formation.

1. Acid hydrolysis (AHY)

63g of the gum powder was weighed into a beaker and diluted with 90ml distilled water to obtain 70%w/v. 20.5 g of concentrated sulphuric acid (corresponding to 10 weight percent of the dry gum Arabic in the stock) was then added to the weighed stock and held at 70°C for four hours with continuous stirring using a regulator hot plate magnetic stirrer.

2. Acetolysis (ACT)

1:1 (weight/weight) ratio of acetic acid and acetic anhydride was thoroughly mixed in a round bottom flask. Thereafter, concentrated sulphuric acid, corresponding to 3%w/v of the mixture was added to the mixture. This mixture was known as the acetolyser.

63g of the gum was weighed in a beaker and diluted with 90ml distilled water. 20.5g of the acetolyser was then added to the stock and the temperature being held at 70°C for four hours, after which the mixture was left to cool to ambient temperature.

3. Acetate formation (AAH)

For this modification, 63g of the dried gum was weighed into a beaker and diluted with 90ml distilled water to obtain 70%w/v. 20.5g of acetic anhydride was then added to the stock. The mixture was held at 70°C for four hours after which it was left to cool to room temperature.

Physical Modification

The physical modification involves mixing the gum Arabic with two different compounds viz: glycerol and ethylene glycol.

1. Glycerol (GLY)

70 weight percent of the gum was prepared in a beaker at 70°C. 20.5g of glycerol was thoroughly mixed with the concentrated stock and allowed to cool to room temperature.

2. Ethylene glycol (EGL)

Ethylene glycol (BDH) was used as received. A similar procedure to that of glycerol was employed for the plasticization of gum Arabic with ethylene glycol.

RESULT AND DISCUSSION

Physicochemical Analysis of Gum Samples

The samples were found to be odorless and tasteless. Their conductivity, total dissolved solid (TDS) and salinity were measured using a conductivity meter. For all the test samples AHY and ACT have the highest values with AHY going extremely high. It records 12.1%, 20.6mS/cm and 11750mg/L for salinity, conductivity and TDS respectively. ACT follows next with 0.3%, 685µS/cm and 335mg/L for salinity conductivity and TDS respectively. The values for all other samples were low as compared to AHY and ACT. This is due to concentration of sulphuric acid used in their modification. The trend for salinity, conductivity and TDS for all the samples is AHY>ACT>AAH>PGM>EGL>GLY>.

The texture of the gums was also observed. PGM, AAH and ACT appeared solid and were ground to powder. EGL was solid but oily due to the nature of ethylene glycol. GLY looked sticky or semi-solid. AHY was completely a dark liquid.

Table 1: Physicochemical parameters of the pure gum as well as the gum modifications.

Parameter	ACT	AHY	EGL	AAH	GLY	PGM
Salinity(‰)	0.3	12.1	0.1	0.2	0.1	0.2
Conductivity(µS/cm)	685	20.6	308	421	261	383
TDS(mg/L)	335	11750	149.8	205	124	177.5
Turbidity	40	485	258	71	68	83
Colour	Buff	black	Ash	jasmine	Brown	White
Texture	Solid	liquid	Solid(oily)	solid	Sticky	Solid
Density(g/cm ³)	0.9944	1.0116	1.0086	1.0113	0.9951	1.0159
M.P(°C)	234		242	229		245
pH	3.78	1.19	4.37	4.1	5.31	4.24

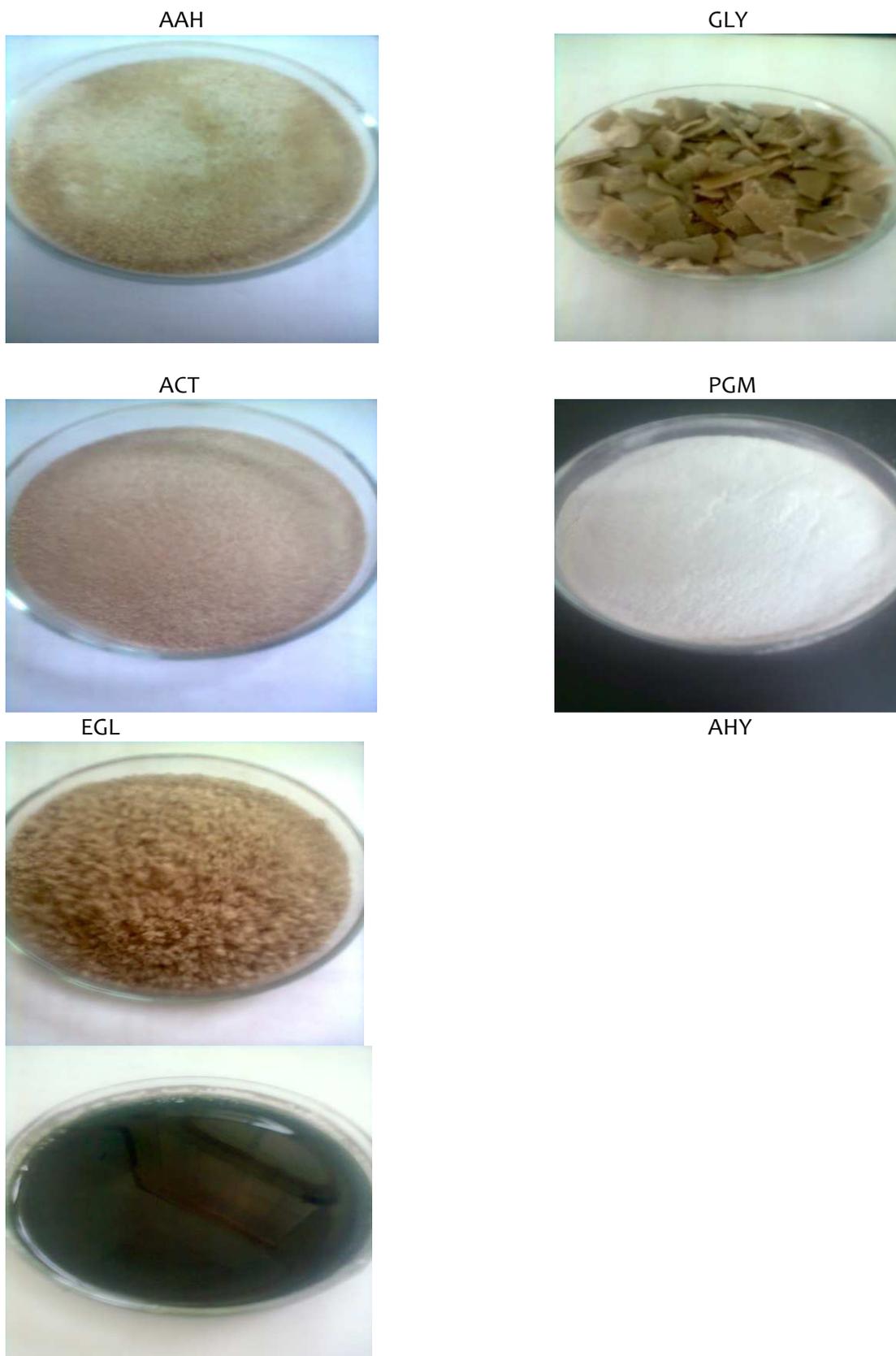


Plate 1: Photographs of the pure gum arabic (PGM) and each gum modification, acid hydrolysis modified (AHY), Ethylene glycol modified (EGL), Glycerol modified (GLY), Acetolysing mixture modified (ACT), Acetic anhydride modified (AAH).

Turbidity is an optical characteristic of a liquid which in general terms describes the clarity of a liquid. The trend for turbidity measurement is AHY>EGL>PGM>AAH>GLY>ACT.

pH is the measure of salinity or alkalinity of a sample. The pH of the pure gum and that of the modifications were quiet below 5.0, except for glycerol modified which was 5.3 at room temperature. This indicates acidic nature, because gums are generally macromolecular acids (Odeku and Fell, 2004). AHY sample is the most acidic with pH value of

1.19. This is due to the concentrated acid used in its modification. The pH value of pure gum Arabic (4.24) is in good agreement with reported pH values for gum arabic and other *Acacia* gums by several authors. The acidity of plant gums is not unexpected since many 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 structures (Abu Baker *et al.*, 2007; Ahmed *et al.*, 2009; Elnour *et al.*, 2009).

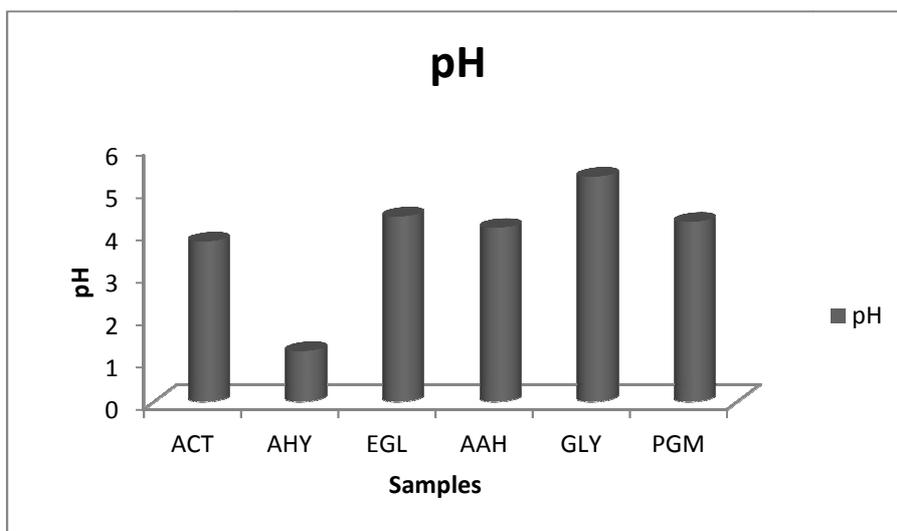


Fig 1: Bar chart showing the pH of the various samples

Melting point analysis of the samples was carried out but the result of AHY and EGL could not be presented due to their nature. AHY was a liquid and EGL appeared very sticky and therefore could not go down the capillary tube. The pure gum has the highest melting point. This could possibly be due to the fact that the modification is believed to have lead to smaller side chains which might enhance chain flexibility. The melting temperature range of 229-245 °C (all samples) suggests that the gum samples were of good thermal stability. However, high melting temperature was likely due to the heterogeneous and polydisperse nature of the unfractionated

gum Arabic exudate, composition-wise (Yusuf, 2011).

Density is the compactness in molecular arrangement in any substance which decides how heavier or lighter that substance is. Density measurement of the samples shows they have higher densities than water. The result indicates that the gum modifications have lower density values than the pure gum sample (PGM). This is not unexpected as the modification of the gums may have caused possible bond scission resulting in the formation of low molecular weight oligomers. Fig 2. shows how densities of the various samples compare with each other.

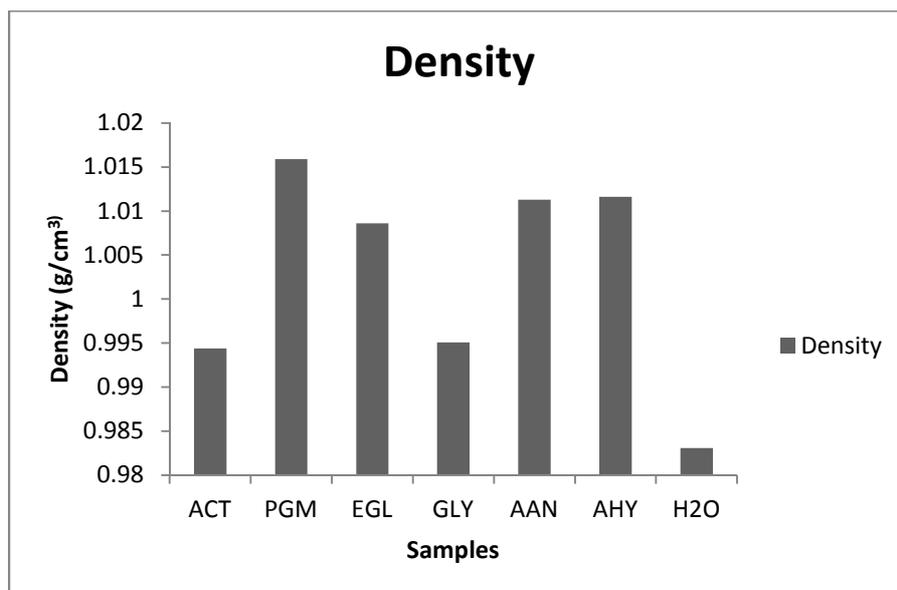


Fig 2: Density of test samples as compared to that of water

Viscosity is a measure of the resistance of a fluid to flow. Viscosity is one of the most important analytical and commercial parameters in polymers because it is affected by the size and shape of macro-molecules. Viscosity of gum solutions at room temperature was found to depend on gum concentration. The viscosities of aqueous dispersions containing different concentrations (2, 1.8, 1.6, 1.4, 1.2, and 1.0 %w/v) of pure gum arabic and that of the modified gums were determined. Table 2. shows comparison between the viscosities of the gums at each concentration. When concentrations were varied for each gum, the viscosities of the solutions increased with increasing concentration. Increase in viscosity

with concentration could probably be due to increasing number of high molecular weight polymeric chains of the gums per unit volume and increased interaction between these chains in aqueous solution or dispersion (Yusuf, 2011). At each concentration the viscosity of AHY is far below that of the other samples, this could be attributed to the fact that acid hydrolysis of polysaccharides normally breaks polysaccharides down to their monomers, dimers and some oligosaccharides (Bockkov and Zaikov, 1979). The products of this hydrolysis are low viscosity liquids. The acid hydrolysis is believed to fragment the molecules by cleaving bonds other than the (1 – 6) which is the most resistant to acid hydrolysis (Guthrie, 1974).

Table 2: Viscosities of the test samples at different concentrations

S/N	Conc.(g/ml)	VISCOSITIES (mpas/sec)					
		PGM	EGL	GLY	ACT	AAH	AHY
1	0.01	26.4	22.8	24	23.4	33.6	13.2
2	0.012	27.6	24	25.2	24	36	14.4
3	0.014	30	24	25.2	24	39.6	18
4	0.016	31.2	25.2	25.8	25.2	43.8	19.2
5	0.018	32.4	25.8	26.4	27.6	46.8	19.2
6	0.02	33.6	27.6	27.6	28.8	50.5	21.6

FTIR OF PURE GUM ARABIC

FTIR spectroscopy has been proved to be a useful technique for characterization of intermolecular interactions between groups in self-polymer or different polymer molecules in blend systems. These intermolecular

interactions, mainly The wave number 605.67cm⁻¹ is a C-H bending vibration of RC=CR alkynes. 705.02cm⁻¹ and 769.63cm⁻¹ are the C-H out of plane vibrations of trans RCH=CHR alkenes. 1157.33cm⁻¹ is typical of C-H wagging of alkyl halides.

The C-H rocking vibration is associated with 1356cm^{-1} . The wave numbers 2727.44 and 2764.09 represent RCHOC-H functional group of aldehydes. The bands 3469.09cm^{-1} and 3553.96cm^{-1} are consequence of the presence of O-H groups. 1715cm^{-1} denote C=O stretch of carbonyls. These functional groups are present in materials like carbohydrates, starch and some other natural gums (Malik et al. 2002).

For the acetolysing mixture modification, several shifts were observed, the C-H bend and C-O stretch at 605.97cm^{-1} and 1004.95cm^{-1} were shifted to 601.81cm^{-1} and 1053.17cm^{-1} respectively. N-O aromatic at 1276.92cm^{-1} was shifted to 1253.77cm^{-1} , N-H out of plane vibration was shifted from 1601.93cm^{-1} to 1630.87cm^{-1} , 1715.74cm^{-1} C=O stretch was shifted to 1734.06cm^{-1} . Isocyanides absorbing at 2153.59cm^{-1} were shifted to 2142.02cm^{-1} while the O-H stretch of alcohols was shifted from 3469.09cm^{-1} to 3471.46cm^{-1} . New bonds were formed at 1397.47cm^{-1} , 2357.09cm^{-1} and 2929cm^{-1} .

In AAH sample the shifts include; C-H bend in alkynes was shifted from 605.67cm^{-1}

to 591.2cm^{-1} . 1004.95cm^{-1} stretch of carboxylic acid was shifted to 1052.2cm^{-1} . N-O aromatics at 1276.92cm^{-1} and N-H out of plane at 1601.93 were shifted to 1253.77cm^{-1} and 1630.87cm^{-1} respectively. A shift was also seen in the absorption frequency of isocyanides from 2153.59cm^{-1} in PGM to 2145.88cm^{-1} in AAH. O-H alcohols also were shifted to 3418cm^{-1} .

The AHY modified gum Arabic shows different absorption frequencies as compared to those of ACT and AAN. In this sample the only shift seen was from 1004.95cm^{-1} to 1053.17cm^{-1} for C-O stretch. Most of the peaks observed are for entirely new bonds. These are 1202.66cm^{-1} , 883.43cm^{-1} , 2946.36cm^{-1} , 1653.05cm^{-1} and 3946.49cm^{-1} .

The FTIR of the modified gums revealed frequencies and peaks due to interaction between the solvents and gum Arabic matrix. The results obtained indicate the presence of several functional groups arising from the formation of new bonds and shifts in frequencies when compared to those deduced from the pure gum Arabic.

Table 3: FTIR data of pure gum (PGM) showing functional group assignment of the various peaks

S/N	PEAK	INTENSITY	AREA	FUNCTIONAL GROUP ASSIGNMENT
1	605.67	44.2101	58.9591	C-H bend
2	705.02	40.0542	35.0379	C-H out of plane
3	769.63	35.5513	54.0166	C-H out of plane
4	1004.95	29.9274	130.0956	C-O stretch
5	1157.33	30.6632	38.4497	C-H wag
6	1276.92	27.5927	82.7976	C-O stretch
7	1356	28.0718	94.9118	C-H rock
8	1601.93	26.7422	75.5933	C=C bending
9	1715.74	30.7319	72.8561	C=O stretch
10	2153.59	47.2996	109.3269	Isocyanides
11	2727.44	29.5151	191.5694	RCHOC-H stretch
12	2764.09	29.5127	33.6713	RCHOC-H stretch
13	2868.24	28.6988	64.7021	-CH ₃
14	2989.76	28.9577	71.0448	C-H stretch
15	3160.47	27.9801	91.0552	OH dimer
16	3308.03	30.1045	93.4743	=C-H stretch
17	3469.09	33.9432	38.3168	O-H stretch
18	3553.96	34.0519	98.4620	O-H stretch
19	3778.68	45.6984	30.3044	

Table 4: FTIR data of acetolysis modification (ACT) showing functional group assignments of the various peaks

S/N	PEAK	INTENSITY	AREA	FUNCTIONAL GROUP ASSIGNMENT
1	420.5	20.577	54.231	
2	472.58	20.322	57.702	
3	601.81	19.609	203.183	C-H stretch
4	1053.17	10.368	290.506	C-O stretch
5	1253.77	16.539	92.564	N-O
6	1397.47	18.172	142.044	C-O stretch
7	1630.87	22.785	76.959	Ar-CH=CHR
8	1734.06	18.403	127.373	C=O stretch
9	2142.02	32.737	164.355	Isocyanides
10	2357.09	29.925	86.751	Si-H
11	2929	17.321	368.2	-CH ₂ -
12	3471.98	10.414	653.785	O-H stretch
13	4001.46	27.893	183.066	
14	4430.64	27.883	168.01	

Table 5: FTIR data of acid hydrolysis modification (AHY) showing functional group assignments of the various peaks

S/N	PEAK (AHY)	INTENSITY	AREA	FUNCTIONAL GROUP ASSIGNMENT
1	443.64	17.616	159.805	-
2	883.43	58.568	35.796	C-H out of plane
3	1053.17	50.085	42.466	C-O stretch
4	1202.66	49.271	105.730	C-O stretch
5	1653.05	53.139	38.0320	C=C stretch
6	1730.21	53.244	80.009	C=O stretch
7	2946.36	49.170	230.190	C-H stretch
8	3412.19	43.150	145.892	O-H stretch
9	3946.49	97.503	2.807	-

Table 6: FTIR data of acetic anhydride modification (AAH) showing functional group assignments of the various peaks

S/N	PEAK	INTENSITY	AREA	FUNCTIONAL GROUP ASSIGNMENT
1	423.390	22.857	61.219	
2	469.680	23.072	33.717	
3	591.200	22.650	205.450	C-Br stretch
4	1052.200	16.149	244.980	C-O stretch
5	1253.770	20.818	82.170	N-O aromatic
6	1407.120	21.270	128.654	Ar C-C stretch
7	1624.120	23.124	86.1880	Ar-CH=CHR
8	1731.170	22.379	116.592	C=O stretch
9	2145.880	32.379	157.250	Isocyanides
10	2929	20.611	442.300	-CH ₂ - stretch
11	3418.940	14.909	576.920	O-H stretch
12	4017.860	28.939	179.920	
13	4350.590	29.003	117.919	
14	4403.630	29.012	54.366	

CONCLUSION

The characterization of the samples shows that the pure gum has the highest density as well as melting point. The pH measurement of the samples revealed that all the gums are acidic having pH values below 7.0. Trend in viscosity measurements at different concentrations indicate that the viscosity increases with increasing concentration of the samples, with AAH sample having highest values for all compositions. AHY samples recorded higher values for conductivity,

salinity and TDS. FTIR spectra of the modified and unmodified samples were also studied. Shifts observed in the modified gums as well as absorption at different frequencies provide evidence for the occurrence of modification of the pure gum Arabic. It is therefore evident that modification of the gum has been achieved, which when used in PVC as an additive could give different mechanical properties, the best of which could be important for industrial purposes.

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