STABILIZATION AND GHARACTERIZATION OF GUM ARABIC DEVELOPED FROM SOLID ACACIA EXUDATE

BY

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NOVEMBER 2005.

DECLARATION

I, AWOYEMI SUNDAY THOMSON, declare that this project is the original

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report of my work and that to the best of my knowledge has never been

presented elsewhere.

All literature cited have been duly acknowledged in the references.

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CERTIFICATION

This is to certify that this project was supervised, moderated and approved by the listed persons on behalf of the Chemical Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, Minna.

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Dr. F. ABERUAGBA HEAD OF DEPARTMENT DATE

DEDICATION

This work is dedicated to the Almighty God for making it possible for me to see the completion of my degree programme and to my Mother and my lovely Wife who are always praying for my success in life.

ACKNOWLEDGEMENT

All praises belong to my heavenly Father for his mercy, guidance and protection throughout my stay in this school, I say thank you Jesus, once again and I shall always be grateful unto you.

To my parents Mr. & Mrs. E.O.Awoyemi, Mr. & Mrs. J.O. Odedeji especially my caring mum, Mrs. M.T. Opaleye, thanks for your moral and financial support and for the constant prayers to see that I achieve the best in life. May God Almighty bless and reward you all abundantly in this world and hereafter.

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I am proud of you because you are so precious to me and I'll remain honest to you, May God helps me.

I will continue loving you, my beloved wife in whom I am pleased.

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ABSTRACT

Stabilization and characterization of gum Arabic developed from solid acacia exudates were carried out by a unifactor simplistic design in which four different samples of gum arabic, each of 20ml were formulated and marked A, B, C, and D. Additives at varying concentrations and quantity were added. The result of the various physiochemical properties such as pH, viscosity, refractive index and specific gravity of different formulated samples A, B, C and D are given below; A(0.86, 0.598, 1.3371, 1.142), B(14.12, 0.870, 1.3452, 1.161), C(14.16, 0.866, 1.3522, 1.193), D(14.20, 0.659, 1.3370, 1.245) respectively. It showed that sample C has the highest viscosity value of 0.870kg/ms at pH 14.12 followed by Sample D with Viscosity value of 0.866kg/ms at pH 14.16 and Samples B and E have viscosity values of 0.598kg/ms and 0.659kg/ms, for pH 0.86 and pH 14.20, respectively. From visual observations, results of samples A, B, C and D after three weeks were comparable to the standard gums.

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NOMENCLATURE

V =	Volume of liquid
T =	Time for a fixed volume of liquid to flow
$L_1 =$	Weight of specific gravity bottle
$L_2 =$	Weight of specific gravity bottle + Weight of Sample
L ₃ =	Weight of Liquid
U =	Viscosity of liquid
SG =	= Specific gravity.

CHAPTER ONE

INTRODUCTION

Adhesives are substances capable of holding material together by surface attachment with the ability to sustain the designed load requirement without deformation or failure.

The great volumes of wood adhesives for structural application are thermosetting phenol-formaldehyde [PF] or urea formaldehyde [UF] polymer or their derivatives while adhesives such as epoxy urethane and poly-vinyl acetate resins are used for interior non –structural application. Glue and other adhesive products were manufactured from hides and bones in the late eighties. The alternative sources of adhesive raw materials were investigated due to the low level of production in this country. It is on this ground that the present work is to develop gum arabic from solidacacia juice.

Gum arabic, also known as gum acacia, is one of the oldest known commercial gums. It is a dried gummy exudate obtained from branches and stems of trees belonging to various species of the genius acacia, (sub-family minosoideae, and family leguminous). The Egyptians juices (by-products) were among the earliest natural polymers to be exploited commercially and still find extensive use today as gum arabic and extended for most wood adhesives.

Gum arabic consists mainly of high- molecular weight polysaccharides and their calcium, magnesium and potassium salts, which on hydrolysis yield arabinose, galactose, rhamnose and glucuronic acid. Items of commerce may contain extraneous materials such as piece of bark, sand, etc which need to be removed before using it.

Gum arabinose is a natural adhesive developed from the solid, acacia by product. It is mostly used as paper gum (walling of paper) and as adhesives for particle board manufacture.

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1.0

1.1 Aims and Objective

The objective of this project was to develop and stabilize gum arabic prepared from acacia juice/ exudates that may be used in paper industries. Also, the physiochemical properties of the adhesive were also investigated.

1.2 Approach

The approach adopted for this work included using unifactor simplistic design

for stabilization and characterization of gum Arabic developed from acacia juice.

CHAPTER TWO

LITERATURE REVIEW

Acacia is the common name for plant of the genus acacia of the legume family, leguminosea. Acacias are known as wattles in Australia and as thorns in Eastern Africa, and are sometimes sold by florists as mimosa in Europe and North Africa.

World climatic zones that have a long, dry winter and a short wet summer often support shrubby vegetation known as thorn scrub and Savannah.

Acacia trees constitute much of the vegetation in such climatic regimes. The trees are characterized by their umbrella shape, with basal branching of the stems; the foliage forms a flattened or curved crown. The flowers usually yellow grow in crowded globes head or cylindrical spites are common, and the Central America bull-horn acacia, acacia Senegal, host a pulp-eating that hollow's out them. Acacia can tolerate long periods of drought and because of the thorns, survive heavily grazed area.

Acacia are used as ornamentals in tropical and sub-tropical gardens, as shade tree and as indoor plants. Livestock are fed from the leaves of some acacias in Australia and some parts of Africa, the seeds or pods of other acacia are eaten by humans.

Australians use acacia wood for railroad ties, wheels, handles and furniture. Some pods yield a substance used for washing silk and as shampoo.

Acacia are also sources of gums, tannin and a dye called clutch.

Various classification of acacia have been made by botanist, there are more than 400 species of acacia, but this research project will be specifically on acacia Senegal, among the acacia species are:

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- (1) Acacia albida
- (2) Acacia compylacant
- (3) Acacia nilotica
- (4) Acacia seyel
- (5) Acacia Senegal

2.0

- (6) Acacia gourmaensis
- (7) Acacia dudgeoni
- (8) Acacia heclii
- (9) Acacia laete
- (10)Acacia macrostachya etc

2.1

History of Acacia Senegal

The small tree, grow mainly in the sub- saharan zone of Africa and it is readily recognized by the triple spines at the base of the bran - chalets.

This character is shared with the very similar acacia dudgeoni, and occasionally a third spine is met within acacia latex. It extends from Senegal to north-eastern Africa and south to Mozambique.

The word "acacia" was derived from the Greek word for thorn. The main source of commercial gum arabic is a single species, Acacia Senegal L.wild, but minor quantities can be obtained from other species. This species of tree is about 4.5-6m high and has a life span of 25-30 years. It grows on poor, sandy, reddish soil. These are some evidences that high yield of gum coincides with poor seasonal rainfall and harsh temperature. The tree is capable of withstanding draught up to 8-11 months in a year and a high temperature up to 43° C.

To obtain the gum, the trees are wounded and the sap allowed flowing out, forming yellowish, transparent lump. Yields can be increased by making incisions in the bark or stripping it from the tree or shrub. Collection of sun dried-dried gum lumps is manual and these are the main harvest during the dry season. Yield per tree does not exceed 300g and no gum is formed during the raining season.

When the trees are in full bloom, Gum arabic is slightly acidic, complex polysaccharide with a high molecular weight of 240,000. It is produced as a mixture of calcium, magnesium and potassium salts (JECFAFAO, 1997). The complex polysaccharide includes: arabinose, galactose, rhamnose or manno-methylose and open chain glecuronic acid.

e.

Gum from Senegal contains 3.8% ash, and 0.34% nitrogen, 0.24% methoxyl, 17% uronic acid and the following sugar constituents after hydrolysis: 45% galactose, 24% arabinose, 13% rhamnose, 16% glucuronic acid and 15% 4-0-methyl, glucuronic acid (Anderson et al).

Three principal fractions have been intensified by hydrophobic affinity chromatography: a low molecular weight arabinogalactan (AG), a very high molecular weight arabinogalactan protein complex (AGP) and a low molecular weight glycoprotein (GI). These components represent 88%, 10% and 1% of the molecule respectively and they contain 20%, 50% and 30% polypeptides respectively. The protein is located on the outside of the AGP unit. The overall conformation of the gum arabic molecules is described by the "wattle blossom" model, in which about fine bulky AG blocks each of 200,000. Da is arranged along the GI polypeptide chain, which may contain up to 1600 amino acid residues (Connolly et al; 1987; Imeson, 1992).

The best grade of gum comes from A. Senegal, which is pale white orange- brown solid, which breaks with a glossy fracture. It is in the form of whole, spheroid tears of varying size with a mattle surface texture when ground, the pieces are paler and have a glossy appearance. Gum from other acacias species may not have the characteristics tear shape and are often darker in colour. Gum from A Seyal is more brittle than the hard tears of A. Senegal. The colours of these exudates also vary widely from pale white to dark brown depending on the species, climate, soils and absorbed impurities. Items of commerce may contain piece of bark and sand which must be removed before using (JECFA 1997).

The main reason for the formulation and exudation of gum by plants is yet to be ascertained and many theories suggested that gum formulation is a protective mechanism resulting from a pathological condition. Evidences about the production of gum arabic support the hypothesis. The acacia trees produce gum arabic only under adverse conditions such as lack of moisture, poor nutrition or exclusively hot weather. The gum is formed in breaks.

The most reasonable explanation however seems to be the simplest among the others, namely that the plant produces the gum in order to seal off the injured part that is primarily to prevent the loss of moisture and not necessarily to prevent the loss of moisture and not necessarily to prevent infection is supported by the fact that gum arabic and gum tragacanth are both produced upon deliberately injuring healthy trees using a small axe to break the outer bark horizontally by making a shallow cut wide. The bark is then stripped from the tree or shrub to form (0.6-1.0m) wounds. The gum collects on the wound within the 3-8weeks depending on weather conditions.

Due to its wide spread use over the centuries, It has also many locally descriptive names such as Kordofan gum from the name of the main production area in Sudan or "hashab" after the local name of the tree in Arabic language, gum Senegal, turkey gum, talha gum and many others. The main producing countries are Sudan republic, which dominates the world production of gum Arabic followed by Nigeria, Senegal, Mali, Chad and Cameroun. World demand is increasing for gum Arabic use as emulsifying, stabilizing and thickening agents in food industries. It is also used in non-food applications such as pharmaceuticals, cosmetics, textiles, lithography and some minor industries (Wang and Anderson, 1994).

2.2

Polysaccharide

Polysaccharides have been described as high molecular weight polymers formed by condensation of many monosaccharide units of derivatives (Dyke, S.F. 1960). They have also been described as polymeric substances, the building blocks of which are monosaccharides (Guthrie, 1974).

From the foregoing, polysaccharide could be said to be long chain carbohydrate molecule built from some monosaccharide such as galactose, rhamnose, glucose etc or their derivatives.

Polysaccharides could be classified based on their chemical compositions. Based on this, polysaccharides which yields only one type of monosaccharide on hydrolysis is called a homoglycan e.g. starch while these yield two or more types of monosaccharide are called heteroglycans e.g. gum Arabic polysaccharides could be classified based on

linearity, (polysaccharides without chain e.g. amylase) and branched (polysaccharides with side chains e.g. gum arabic)

2.2.1 Linear neutral polysaccharides

All polysaccharide are hydrophilic, which is to say that in the presence of water they combine with water molecules to bring about a partial immobilization of the near molecules. This in water, the polysaccharide may completely surround itself with an atmosphere of partially immobilized water molecules.

If the polysaccharides molecule dissolves, this atmosphere of water molecule is carried with it. Associated water molecules are held mainly by hydrogen bonds however, other secondary forces, such as ionic changes and dipole and induced dipole interactions, may also be involved.

The atmosphere of associated water molecules may cause the linear molecule to dissolve or, if not, it may help to maintain solubility after dissolution is brought by other means such as with strong alkali. Thus, the polysaccharide molecule in water will appear to occupy a larger space due to its appearance enlarged diameter. As this extended molecule, with its apparently enlarged cross-section, gyrates by reason of thermally induced, but uneven, impacts of the ambient solvent molecules, it effectively sweeps through a large roughly spherical volume. This large space requirements means that, at very low concentrations, extended polysaccharide molecules will come into contact with each other and give the solution a significant viscosity which will increase rapidly with concentration. Linear polysaccharides will occupy more space and, therefore, show greater viscosity than highly branched polysaccharides of equal molecular weight.

Linear polysaccharides as they gyrate and flex in solution due to Brownian impacts, frequently collide over a portion of their length, shearing off associated water molecules and forcing segments of the two chains sufficiently close that secondary inter molecular forces cause the two micro molecules to attract each other and adhere in this area. Further flopping of the chains works larger sections together until the molecules have united over much of their lengths. Thus, in solutions, particles of associated

molecules quickly form and grow through colloidal size by deposition of further polysaccharide molecules or by coalescence of several particles. Soon the particles enlarge beyond colloidal size to where it is too large to remain in suspension and precipitation occurs.

Due to their strong powers for adsorption, linear neutral polysaccharides are good coating agents for textiles and paper. Like other linear molecules, they usually can be cast into self- supporting sheets and may have use as films or as packaging materials. In general, linear neutral polysaccharides are not tacky.

2.2.2 Branched neutral polysaccharides

Highly branched neutral polysaccharides produced much less viscosity in aqueous dispersion than linear polysaccharides of equal molecular weight. However, branched molecules are equally fully hydrated, that is, surrounded with an atmosphere of associated water molecules. When limbs of these molecules collide in solution they may entangle, if the concentration is high enough, so that a gel results. Extensive association cannot form since it is not possible for the bush-like molecules to fit closely upon each other. Hence, solutions of branched molecules are stable and do not undergo spontaneous precipitation as do solutions of linear molecules.

Partially, because of entangling with one another in solution, branched polysaccharides can produce tacky pastes if concentrations are sufficiently high.

When structure of branched polysaccharides are dried, the molecules still do not form strong linear molecular associations, hence, if water again added to the dry coating, the polysaccharide easily dissolves or reconstitutes, softening quickly to a tacky layer.

This property makes many of the branched polysaccharide useful as remoistening gums or adhesives. Highly branched polysaccharides, like other highly branched polymers form only brittle films and consequently have no application as self supporting sheet.

Some polysaccharides are long chains with numerous very short branches. These molecules have many of the properties of both linear and highly branched molecules.

Good example, are guar gum and locust bean gum. The former, guaron is a long chain of D-mannose units with single D-galactose units joined to alternate units of the main, chain. As a long linear like polysaccharide, its solution is highly viscous. However, the D-galactose side groups fend off other colliding molecules and prevent the associative lining up of molecules as would occur in solutions of purely linear molecules. Therefore, guar gum solutions are stable when its solution are dried or when the polysaccharides are precipitated from solution, extensive inter molecular association is prevented and redissolution occurs rather readily.

2.2.3 Polysaccharides with strong acid groups

Polysaccharides with numerous strong acid groups attached as highly hydrated, bear ionic charges and hence are both highly viscous and stable in solution. Examples are certain polysaccharides derived from seaweeds, such as furcellaran and caurageenan. which are esterified with sulphuric acid to the half ester form. Ionization of such strongly acidic groups are not repressed extensively at low pH levels, so the polysaccharide molecule maintains its ionic charges with their high degree of hydration and is stable in solution of low pH.

2.2.4 Polysaccharides with basic groups

No naturally occurring polysaccharides containing basic groups is sold commercially, but work on chitin shows signs of incipient application. A linear basic molecule, such as deacetylated chitin, is soluble in strong acids and shows particularly tenacious absorbance on negative surfaces. Such a molecule in solution remains extended. It will combine in solution with cations polysaccharides or other negative polymer molecules to produce a precipitate by mutual neutralization and aggregation.

2.3

Modified Gums

Chemical modification of polysaccharides is useful for alteration of chemical and physical properties to give the polysaccharide new applications. In general, the properties of neutral polysaccharides are altered to a remarkable degree by the introduction of very small amounts of substituent groups of either neutral or ionic type.

Introduction of neutral groups

Substitution of neutral groups on linear polysaccharides leads to increase solution viscosity and stability. In effect substitution of methyl, ethyl, hydroxyethyl, and similar groups tends in a broad sense to give linear polysaccharide the behaviour patterns of guar or locust bean gums.

Solubility characteristics are enhanced more by the use of hydroxylated substituent groups, such as methyl or ethyl. Water solubility diminishes and solubility in organic solvents increases as hydrophobic groups are introduced a D.S of about 1.0.

2.3.2 Introduction of basic groups

2.3.1

Basic groups are introduced directly into the sugar rings of polysaccharides with great difficulty because it is hard to find a convenient reaction by which the hydroxyl groups are replaced with amino groups. On the other hand it is quite easy to derive a polysaccharide with a group which contains a free or substituted amino group. It is also possible to react an ester of a carboxylated polysaccharide with ammonia to form the amide.

2.3.3 Other chemical modifications of polysaccharides

Other methods of chemical modification of neutral polysaccharide include thermal dextrinization, partial hydrolysis and mild oxidation. Control of properties through proper choice of kind and amount of substituent groups is quite well known.

2.4 <u>Chemistry of Gums</u>

Gums are uncrystallizable and are usually composed of C, H and O. They are water loving colloids; they may apparently dissolve, but actually disperse or swell or absorb water to form commercially valuable substances. The gums have the characteristics property of forming in water or by absorbing many times their own volume of that solvent.

Gums in general swell in water to form viscous solution. The affinity for water exhibited by gum arabic is not the characteristic of all the other gums; nevertheless water is the solvent common for all the gums. The viscous solutions obtained for the

gums were colloidal in behaviour, exhibit swelling pressures, and form gel structures at extremely low concentration and over a wide concentration range.

The gums consist primarily of compounds of C, H and O of the types related to the starches and sugars (Carbohydrates). The carbohydrates, although composed of only three elements are very numerous owing to the differences caused by the spartial arrangement of the atoms in the molecules.

Glucose is one of the monosaccharides, in this same group we find sugar arabinose, $C_5H_{10}O_5$, whose chemistry evolved through the study of the hydrolysis of gum arabic. Arabinose may also be derived from tragacanth, ghatti, and other tree exudation gums.

Arabinose contains 5 carbon atoms and is often referred to as pentose



Often associated with arabinose is a hexose sugar galactose



Galactose is found in locust bean gum as well as in tree exudation gums, while arabinose and xylose, a pentose are found together in seed extracts such as flax seed, quinee and psyllium.

The structure of xylose is given as

Mannose another hextose, is found with galactose in locust bean, mannose is given as

СНО HO--C- H HO--C- H H --C- OH II --C- OH I --C- OH

Structure of Gum Arabic

2.5

The exact structure of gum arabic is not known, but it exists in a nature as a neutral or slightly acidic salt of a complex polysaccharide containing Ca, mg and K salts with a molecular weight of about 240,000-300,000.

It is known that hydrolysis of gum arabic yield four sugars which make up to the basic gum molecules: L- arabinose, L-rhamnose, D-galactose, D-glucuronic acid. The glysocidic bonds vary. Thus, auto hydrolysis of Arabic acid in water (pH=2) gives 34.4% of L-arabinose, 14.2% of L-rhamnose, 3-0-X-D- galactose pyranosyl-Larabinose and degraded gum arabic.

When mineral acid is used for further hydrolysis, there is obtained 42.1% of D-galactose, 15.5% of D- glucuronic acid and some 6-0 (B-D- glucopyranosyluronic acid) –D- galactose.





2.6 Physical Properties of Gum Arabic

The physical properties of gum arabic are of utmost importance in determining their uses, commercial value and their end use. Among the factors that can affect the physical properties of the gum when collected that is the length of time it has remained to the tree after secretion. The treatment given to the gum after collection, such as washing, drying, bleaching in the sun and storage temperature. Gum collected from the same plant at different season of the year, from the same species but from plants growing under different climatic and adaptic conditions would have differences in their physical properties.

Gum Arabic is exuded in a variety of shape and forms the best known being the tear-drops and globular shape of various grades. The surfaces of most gums are perfectly smooth when fresh but becomes rough or covered with crack when dry. The surfaces of most gums are perfectly smooth when fresh but becomes rough or covered with crack when dry. The colours of gum Arabic varies from pale while through various shades of yellow, amber, and orange to dark brown. Gum that contains no bark or foreign matter and is odourless, colourless and tasteless is the best grade. It does not affect the odour, colour or taste of the system in which it is used.

Gums vary in hardness depending on the moisture contents, which ranges from 12-16%. Density also proves variables in gums due to the amount of air that may have become incorporated with it when it was formed from the source.

2.7 <u>Chemical Properties of Gum Arabic</u>

Gum arabic as the calcium, magnesium and potassium salt of Arabic acid reacts with many reagent in a manner similar to that of other polysaccharide acid salts. Solution of gum arabic will produce precipitates or heavy gels on addition of any of the following reagents, potassium silicate, sodium silicate, million reagents. In general, trivalent metallic salts will cause precipitation of gum arabic.

Upon treatment with dilute acids, gum arabic is hydrolysed to yield a mixture of L-arabinose, L-rhamnose, D-galactose and an aldobiouronic acid composed of Dglucuronic acid and D- galactose. With nitric acid, treatment, gum arabic yields mucic, saccharic and oxalic acid.

2.8 Quality Parameters for Gum Arabic

2.8.1 Viscosity: Gum arabic is unique in that it is extremely soluble and not very viscous at low concentrations, whereas most other gums form highly viscous solutions at low concentrations of about 1-5%. High viscosities are not obtained with gum arabic until concentrations of about 40-50% are obtained. This ability to form highly concentrated solution is responsible for the excellent stabilizing and emulsifying properties of gum viscosity.

2.8.2

Effect of pH .

The effect of pH on gum arabic solutions has been reported by several investigations that tend to agree that Arabic acid is a strong monobasic acid (Taft et al, 1931); (Thomas et al, 1928).

The viscosity of gum arabic rises sharply with increasing pH maximum at about pH of 5-7, then falls slowly to about pH of 10-14. Solutions of gum arabic are normally slightly acidic having a pH of about 3.9- 4.9.

Polarization

Force acts between different parts of the same molecules and between polymer and solvent used.

These forces include hydrogen bonds, ionic charges, Van der Waal forces, dipoledipole attractions. All of these forces affect such properties of gum as gel forming tendency, viscosity and adhesiveness.

2.8.4

Rheological Behaviour

Gum arabic solution exhibit typical Newtonian behaviour at concentration up to 40%. About 40% solution takes up pseudo plastic characteristics as denoted by a decrease in viscosity with increasing shearing stress (Aranjo, 1966).

2.8.5

Solubility

Among hydrocolloids, gum arabic is unique because of its very high solubility in water. It can form solution up to 50% concentration whereas most gums are limited to solubility of less than 50%.

This property makes it very useful in application where low viscosities are required.

Gum arabic solutions are slightly acidic with pH of 4.5-6.3. The maximum viscosity of the gum solution is at pH of 6.7 and is lowered by the addition of electrolytes or long exposure to acid conditions which may cause partial hydrolysis. It is insoluble in alcohol and other organic solvents but soluble in aqueous ethanol up to about 60% of alcohol.

2.9

Uses of Gum Arabic

2.9.1

Food and drinks industries

Gum arabic is also used to modify the consistency of certain foodstuffs by increasing moisture holding capacity, for example, it is used as a gelling agent in canned meat and fish, marshmallows, jellied carries and fruit jellies. The properties of gum arabic as a stabilizer and thickener also help to keep solids dispersed in chocolate milk, air in whipped cream and of facts in manufacturing salad dressing. Gum arabic is also an ingredient in some preparations used for wine clarification.

Adhesives

The industrial applications of gum arabic adhesives at present are very limited, though small quantities go to every country in the world where stamps are made. It is also been used as an adhesive medium in wrapping paper and binding cement for iron casting.

2.9.3

Cosmetics

In the cosmetic industry, gum arabic has limited applications on a wide range of products. In lotions and protective cream. It stabilizes the emulsion, increases the viscousity adds a smooth feel to the skin and forms a protective coating.

2.10

Stabilization of Gum

Stabilization could be defined as any process that minimizes or prevents fluctuations in the quality or condition of a material that is, any substance that tends to maintain the physical and chemical properties of a material. Degradation that is reversible changes in chemical compositions or structures is responsible for the premature failure of materials.

Preservatives or stabilizers are often used as to extend the useful of life to materials as well as to maintain their critical properties above the design specification. Forces act between molecules, between different parts of the same molecules and polymers and solvent. These forces include bonding, ionic charges, dipole and induced dipole attraction and Van-Der Waal forces.

All these affect such properties as gel forming tendency, viscosity and adhesiveness, type of linkage due to their effects on chain flexibility are important also in determining physical properties. For example, it is known that linear molecules make more viscous solutions than Long Branch molecules of similar molecular weights but they have a tendency to precipitate because of association of the chain. If this association is prevented, stability can be achieved without much sacrifice of viscosity. All these conditions can be stabilized or accomplished with preventive or chain breaking antioxidants. Preventive antioxidants stabilizer reduces the number of radicals

formed in the initiation step.

3.0 <u>METHODOLOGY</u>

The equipment and materials used during the experiments are given in

Tables 3.1 – 3.2:

Table 3.1 The Equipment Used for the Experiment

Equipment	Sources.	Research Code	Comment
Viscometer	Pyrex, England	PE 101869	U-tube glass apparatus
pH meter	Kent Ind. Ltd,	KE 896	Digital display
	England		
Refractometer	England	BS 517	Electrical
Magnetic Stirrer	Ohaus, UK	N.A	Stainless Steel

Table 3.2 The Materials Used for the Experiment

Materials	Sources	Research Code	Comment
Sodium Metaborate	BDH Chemicals	N.A	Pellet
Gum Arabic	Acacia Exudate,	N.A	Solid
Sample	Minna		

3.2 Preparation of Additives

(A): Solution of calcium chloride (CaCl₂): it was prepared by dissolving 5g of CaCl₂ in
100ml of distilled water.

(B): 50% w/v of sodium hydroxide (NaOH) was prepared by dissolving 50g of NaOH in 100ml of distilled water.

(C) Solution of Acidified potassium permanganate (KMnO₄): This was prepared by dissolving KMnO₄ in 50ml of distilled water and 20ml of conc.H₂SO₄ acid was added, the solution was made up to 100ml by further addition of distilled water.

Gum arabic was dissolved in hot water in ratio 1.75 to 1.5 respectively to have a highly viscous solution and was filtered using a 500μ m sieve to remove dirt and tree barks so as to get a clear gum solution.

The filtrates were distilled and its viscosity, pH, refractive index, SP.G. was determined. It was now marked "Raw gum".

Four different samples of the raw gum each of 20ml were taken and marked A, B, C, D.

Additives prepared were added, and their concentrations were varied in other to determine their effect on the adhesive properties as well as to obtain the best formulation. The variation in the additives is shown in table 1.0. The addition of the various reagents was followed by continuous stirring for thorough mixing and various results were obtained and presented in Chapter four.

TABLE 3.3	Formulation of	Gum Arabic using	Unifactor	Simplistic Design
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Sample of Gum Arabic	**************************************	Α	В	С	D	······
Sodium Hydroxide (NaOH) (ml)		4	8	12	16	
Sodium Hydroxide+ Sodium Metaborate (ml)			12	16	4	
			en de la composition de la composition La composition de la c			
Calcium Chloride (CaCl ₂) (ml)		12	16	.4	8	

Acidified Potassium permanganate (KMnO₄) (ml). 16 4 8 12

CHAPTER FOUR

4.0

4.1

RESULTS and DISCUSSION

Results

Table 4.1: Physiochemical Properties of Samples

Properties	Sample A	Sample B	Sample C	Sample D	SampleE
pH at 30°C	4.40	0.86	14.12	14.16	14.20
Viscosity (kg/ms)	3.830	0.598	0.870	0.866	0.659
Refractive Index	1.9210	1.3371	1.3452	1.3522	1.3370

The samples A, B, C. and D were observed for three weeks and characterized again to know the effect of ageing on the produced gum Arabic.

3.4 Characterization of Gum Arabic

3.4.1 Determination of refractive index

Alcohol and distilled water was used to clean the prism assembly cover and desired position was obtained by turned mode selector. Glass dropper was used to apply sample solution to the measuring prism surface.

The prism cover was closed and the exposed face was fully illuminated. Shadow lines at the bottom of the field of view were position by rotating adjustment controlled clockwise. The eye piece was rotated to bring the crosshair into focus. The adjustment control knob was turned so that the shadow lines were centered at the crosshair. The read button was obtained when mode selector switched to the refractive index was turned.

3.4.2

Density (Specific gravity)

The empty specific gravity bottle of volume 25ml was weighed and recorded. It was then filled with gum arabic sample was recorded. The weight difference between the filled bottle with gum Arabic and empty bottle equaled the mass of gum Arabic, hence density of samples A. B. C and D were calculated.

3.4.3

Determination of pH

The pH of the sample was determined by using pH meter. Buffer solutions were used to standardize the pH meter so as to measure accurately.



These values show that sample A i.e. raw gum has pH of 4.40 which is acidic in nature and the natural pH of gum from acacia Senegal is 3.9-4.9 (Anderson et.al; 1990).

Sample B has a higher acidity because of the quantity of acidified potassium tetraoxomanganate (V11) solution was higher, while samples C, D and E are alkaline because the quantities of the base additives were higher than the amount of acidified KMnO₄ solution.

The viscosity was found to be 3.83kg/ms, 0.598kg/ms, 0.870kg/ms, 0.866kg/ms and 0.659kg/ms for samples A, B, C, D and E respectively.

Sample A which is the raw gum has a viscosity value of 3.83kg/ms and a pH of 4.40 which is within the range at which maximum viscosity of gum arabic in solution occurs at pH 4.5-5.5 (Glicksman et al. 1973), lowering of viscosity and interfacial tension of gum arabic produces favorable emulsifying agent, its stabilization of emulsion is dependent mainly on the coherence, rigidity, elasticity and adsorption of the interfacial film (Glicksman et al, 1973).

Also, the low viscosity of Sample B is due to the quantity of calcium chloride added, where it has the resultant effect of reducing viscosity of gum arabic. Also, drop in viscosity is due to the increase in pH of the sample towards being more acidic.

Sample C has the viscosity value which means it is more viscous because it contained the lowest quantity of $CaCl_2$ and $CaCl_2$ has the effects of reducing and increasing the viscosity as its concentration increases.

The fall in viscosity from 0.870kg/ms for C, for 0.866kg/ms for D and 0.659kg/ms for D due to the increasing alkalinity because viscosity reduces as alkalinity increases (Glicksman et al, 1973) where samples C, D and E have pH values of 14.12, 14.16 and 14.20 respectively.

The specific gravity values of samples A, B, C, D and E are 1.260, 1.142, 1.161, 1.193 and 1.245 for samples A, B, C, D and E respectively. It can be seen that the raw gum A, was denser than the remaining samples to which additives were added to i.e. B, C, D and E.

CHAPTER FIVE

CONCLUSION and RECOMMENDATIONS

5.1

5.0

5.2

Conclusions

The following conclusions can be drawn from the unifactor simplistic formulation; that sample A-E had viscosity which are comparable to the literature values, with the highest being sample C which has viscosity value of 0.870kg/ms, pH value of 14.12, specific gravity and refractive index of 1.161 and 1.3452, respectively.

Also, from the above comparison and based on visual observation of the samples, sedimentation occurred in samples B, D and E after three weeks but sample C showed no such sign.

Therefore, it can be concluded that sample C possessed best quality among the five different samples because of its high viscosity value and absence of sedimentation.

Recommendations

(i) Hot plate with a magnetic stirrer and temperature variations should be used for heating the gum Arabic solution instead of using a hot plate with a magnetic stirrer and inserting mercury in glass thermometer that causes temperature flunctuations.

(*ii*) To improve the stability, other ranges of stabilizers can be tried during the formulation.

APPENDIX CALCULATION OF VISCOSITY A1 Viscosity was calculated by using the formula below: μ = 8 Π t Where Π =22/7. t =time taken for a fixed volume of liquid to flow. For sample \underline{A}_{i} t = 152.33sec. $\mu = 8* 22/7 * 152.33 = 3,830.01$ Cp To convert from Cp to kg/ms, $1Cp = 1 * 10^{-3} \text{ kg/ms}$ 3,833.0kp = $1*10^{-3}/1*3.830.01 = <u><math>3.830$ kg/ms</u>. For Sample B t = 23.78 sec, $\mu = 8*22/7 * 23.78 = 597.8$ cp. But $1c.p = 1 * 10^{-3} \text{ kg/ms}$ $(597.8*1*10^{-3})$ kg/ ms = 0.598 kg/ms. For Sample C t = 34.63 sec, $\mu = 8*22/7 * 34.63 \text{ sec} = 870.34 \text{Cp}$ But 1 Cp = $1 * 10^{-3}$ kg/ms. 870.34Cp = (1* 10^{-3} * 870.34) kg/ms = 0.870kg/ms For Sample $D_{1}t = 34.44$ sec. $\mu = 8 * 22/7 * 34.44 \text{ sec.} 865.7 \text{Cp}$ $865.7Cp = 865.7 * 1 * 10^{-3} = <u>0.866kg/ms.</u>$ For Sample \underline{E} t = 26.24sec $\mu = 8 * 22/7 * 26.24 = 659.483$ Cp $1Cp = 1 * 10^{-3} kg/ms.$ 658.729Cp = 1 * 10^{-3} * 658.729 = 0.659kg/ms. CALCULATION OF SPECIFIC GRAVITY A2: Sample A Weight of Specific gravity bottle + Lid, $L_1 = 9.48g$ Weight of Specific gravity bottle + Weight of Sample, $L_2 = 40.98g$ 27

$$S.G = (L2 - L_1) / V = (40.98 - 9.48) / 25 = 1.260$$

Sample B

Weight of Specific gravity + Weight of Sample, $L_2 = 38.03g$

$$S.G = (L_2 - L_1) / V = (38.03 - 9.48) / 25 = 1.142$$

Sample C

Weight of Specific gravity + Weight of Sample, $L_2 = 38.51g$

$$S.G = (L_2 - L_1) / V = (38.51 - 9.48) / 25 = 1.161$$

Sample D

Weight of Specific gravity + Weight of Sample, $L_2 = 39.31g$.

$$S.G = (L_2 - L_1) / V = (39.31 - 9.48) / 25 = 1.193$$

Sample E;

Weight of Specific gravity + Weight of Sample, $L_2 = 40.61g$ S.G = $(L_2 - L_1) / V = (40.61 - 9.48) / 25 = 1.245$ The refractive index of the raw gum A was more than the Samples B, C, D and E which are: 1.3371, 1.3452 and 1.3370.

For visual observation made after three weeks: Sedimentation occurred in samples A, B and D after three weeks which did not occur in sample C. Also, colour changed in all the samples was due to the bleaching action of acidified KMnO₄ which made colour changed in A more pronounced because of the high quantity of KMnO₄ added.

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