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PREPARATION OF CALCIUM GLUCOURONATE FRPM ACSCIA SEYAL VAR. SEYAL GUM

Dissertation submitted in partial fulfillment of the Regiment of the Degree of B.S.C in Chemistry

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September2016

الآية

بسم الله الرحمن الرحيم

قال تعالى: {والَّذِينَ يَكْنِزُونَ الذَّهَبَ والْفِضَّةَ ولا يُنفِقُونَهَا فِي سَبِيلِ اللَّهِ فَبَشِّرْهُم بِعَذَابٍ أَلِيمٍ (34) يَوْمَ يُحْمَى عَلَيْهَا فِي نَارِ جَهَنَّمَ فَتُكْوَى بِهَا جِبَاهُهُمْ وجُنُوبُهُمْ وظُهُورُهُمْ هَذَا مَا كَنَزْتُمْ لأَنفُسِكُمْ فَذُوقُوا مَا كُنتُمْ تَكْنِزُونَ}

سورة التوبة الآية 34–35

صدق الله العظيم

صدق الله العظيم

DEDICATION

- To each of the following in the presence of God and his messenger
- To the fountion of patience and optimism and hope.

Our mother

To the big heart

Our father

To those who have demonstrated to me what is most beautiful of life

Our brothers

To the people who paved our way of science and know ledge all our

All our teachers

To the taste of the most beautiful moments with

Our friends

We guide this research

Acknowledgements

All thanks to almighty Allah for giving me health and patience to accomplish this work.

I would like to express my deepest gratitude and respect to my supervisor Dr. Mohammed Elmubark Osman for helping me during this study and providing me extremely useful discussion and for his friendly guidance throughout this work.

مستخلص البحث:

تمت دراسة الخواص الفيزيوكيمائية لصمغ الطلحة ووجد أن نسبة رطوبة العينة 10.14% ومحتوى الرماد الكلي 3.42% والرقم الهيدروجيني 5.53%

تم قياس الرقم الهيدروجيني بعد عملية التبادل الأيوني وبعد إضافة كربونات الكالسيوم ووجد أن قيمة الرقم الهيدروجيني (3.80 ، 11.20) على التوالي كما تم تقدير بعض العناصر بعينة الطلحة عن طريق جهاز الامتصاص الذري ووجد أن نسبة الكالسيوم 1815ppm ونسبة البوتاسيوم 0.0793ppm.

Abstract:

Acaia seyal var. seyal gum sample was authenticated by evaluating its physio chemical properties, the results show that the moisture content was (10.14%). Total ash was (3.42%) pH was (5.53%), some elements

pH were determined after ion exchange and after added calcium carbonate and pH was (11.40 – 3.80) respectively

Were determined using Atomic Absorption, the ratio of calcium element was found (1815), and the ratio of potassium element was fund (0.0793).

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Introduction and Literature Review

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Introduction and Literature Review

1.Introduction:

Gum Arabic, also known as *Acacia* gum, is a natural gum made of the hardened sap of *Acacia Senegal var seyal* trees. Originally, gum Arabic was collected from *Acacia seyal var seyal,* in the present day, gum Arabic is, predominantly, collected from *Acacia Senegal var seyal* Producers harvest the gum, proposes from wild trees, mostly within Sudan gum and throughout the Sahel, from Senegal to Somalia—though it is historically cultivated in Arabia and West Asia.

Gum Arabic is a complex mixture of glycoprotein's and polysaccharides. It is the original source of the sugars arabinose, galactotose, rhamnose, which were first discovered and isolated from it, and are named after it.

Gum Arabic is used, primarily, in the food industry as a stabilizer. It is edible and has anumber E414. Gum Arabic is a key ingredient and emulsifier in traditional lithography and is used in printing, paint production, glue, cosmetics and various industrial applications, including viscosity control in inks and in textile industries, though less expensive materials compete with it for many of these roles.

While gum Arabic is now produced throughout the African Sahel, it is still harvested and used in the Middle East. For example, Arab populations use the natural gum to make a chilled, sweetened, and flavored gelato-like dessert.

1.2 Literature Review :

1.2.1 Acacia seyal var. seyal tree :

Acacia seyal is a small to medium-sized tree, growing to 17 m tall and 60 cm in diameter at breast height; crown is umbrella shaped, resembling that of A. tortilis var raddiane, var tortilis and var spirocarper. A characteristic feature of the tree is its rust-coloured powdery bark; *A.seyal var. fistula* has whitish bark. Large, straight spines occur on the branches, and smaller, curved thorns are present near the tips of the branches.

1.2.2 Botanical Classification of *Acacia seyal Tree*:

Family : *Leguminous*

Sub Family: *Mimosoideae*

Genus : *Acacia*.

Species : s*eyal var*. f*istula*

s*eyal var*. s*eyal*

Vercular surnames : t*alha*

1.2.3 Definition

Gum Arabic was defined by the Codex Committee for Food Additives, 1999, as the dried exudates from the trunks and branches of *Acacia senegal* or Vachellia (Acacia) seyal in the family Leguminosae *(Fabaceae).*

1.2.4 physical properties of Acacia gum :

The physical properties of gum Arabic, established as quality parameters include moisture, total ash, volatile matter and internal energy. Gum Arabic is a natural product complex mixture of hydrophilic carbohydrate and hydrophobic protein components . Hydrophobic protein component functions as an emulsifier which adsorbs onto surface of oil droplets while hydrophilic carbohydrate component inhibits flocculation and coalescence of molecules through electrostatic and steric repulsions in food additives .(Montenegro 2012)

Moisture content facilitates the solubility of hydrophilic carbohydrates and hydrophobic proteins in gum Arabic (Elmqvist, 2003). Total ash content is used to determine the critical levels of foreign matter, acid insoluble matter, salts of minerals composition.

The cationic compositions of ash content are used to determine the specific levels of heavy metals for safety of gums.

Volatile matter of gum Arabic determines the characteristics and the degree of polymerization contained in sugar compositions (arabinose, galactose and rhamnose) which exhibits strong emulsifying properties functioning as binders and stabilisers in the making of cough syrups in pharmaceutical industry . Internal energy of gum Arabic is the actual energy required to produce the amount of carbon when the gum is heated to 500°C to release carbon dioxide gas. Optical rotation is used to determine the nature of sugars in gum Arabic obtained from A. senegal

variety senegal. The specifications state that the best quality of gum Arabic must have negative optical rotation with the range of -26° to -34° (Table 1). Nitrogen content in gum Arabic determines the number of amino acid, (protien), compositions, with the range of 0.26 to 0.39% (FAO, 1990).

Gum Arabic is used as an emulsifier and stabilizer in the food and pharmaceutical industries (Osman et al.,1993a, b). Other industrial products that use technical grades of gum Arabic include adhesives, textiles, printing, lithography, paints, paper sizing and pottery glazing (Idris et al., 1998). Gum Arabic is produced from natural stands of A. senegal varieties in arid and semi-arid lands (ASAL) ecosystem of northern Kenya (Chikamai and Gachathi, 1994; Chikamai, 1997). Gum Arabic is collected during the dry seasons by herdsmen and women groups (pastoralists) from differ-rent botanical sources. The harvested gums are mixed and sold to middle businessmen in local trading centres who export with standard quality control to world market (Chikamai and Gachathi.

1.2.5 Chemical properties and structure of *Acacia seyal* gum:

Acacia gum, generally, consists of a group of macromolecules characterized by high proportion of carbohydrates which are, predominantly, composed of D-galactose and L- arabinose units and proportion of proteins, these composition may vary slightly depending on its origin climate harvest season and tree age.

Chemistry of gum Arabic:

The structure of gum Arabic *(Acacia seyal)* has been studied using methyration analysis and 2D (cosy, tocsy, HMQC and HMBC) NMR spectroscopy.

Galacturonic acid (13.66%) is major component not previously, identified. The backbone is made up of 1.3 – linked spectroscopy galactopyranosyl (Galp) residues of substituted at 0-2, 0-6, or 0-4 positions, which are terminated with mainly arabinofuranosyl (A rat) galactopyranosyl (galpa), rhamnaopyranosyl with arabionpyionyl (ARAP), and glucuronopyranosya (GlcpA) residues there are long side chains of ⟶ 3) –a – L – Araf – (1 → and 2) –a –L - Araf – (1 ⟶ linkad to the backbone.(lelon 2010)

Proposed structure of the carbohydratein Acacia seyal – R is are of these following residues: T- Rhapl⟶ , T-L Araf1, →T-L –Arap. 1⟶ TGICPAI, → GalpAI⟶ , T-L-Araf1.⟶ 3-L- Araf1. ⟶ ,2-l- Araf1 ⟶ the galactic moieties are in B – D form with the galactuaud , are bingos and rhamnoseli a-L form . (dauqan, 2013)

1.2.6 Usaes:

Gum Arabic's mixture of polysaccharides and glycoprotein's gives it the properties of a glue and binder that is edible by humans. Other substances have replaced it where toxicity is not an issue, as the proportions of the various chemicals in gum Arabic vary widely and make it unpredictable. Still, it remains an important ingredient in soft drink syrups, "hard" gummy candies such as gumdrops, marshmallows, M&M's chocolate candies—and edible glitter, a popular modern cake-decorating staple. For artists, it is the traditional binder in water color paint, in photography for gum printing, and it is used as a binder in pyrotechnic compositions. Pharmaceutical drugs and cosmetics also use the gum as a binder, emulsifying agent, and a suspending or viscosity increasing agent. Wine makers have used gum Arabic as a wine fining agent.

It is an important ingredient in shoe polish, and can be used in making home made incense cones. It is also used as a lickable adhesive, for example on postage stamps, envelopes, and cigarette papers. Lithographic printers employ it to keep the non-image areas of the plate receptive to water.(Glicksman, 1983) This treatment also helps to stop oxidation of aluminium printing plates in the interval between processing of the plate and its use on a printing press.

Also called acacia after the original source, gum arabic is used as an emulsifier and a thickening agent in icing, fillings, chewing gum and other confectionery treats.

Gum Arabic is used as a binder for water color painting because it dissolves easily in water. Pigment of any color is suspended within the acacia gum in varying amounts, resulting in water color paint. Water acts as a vehicle or a diluents to thin the water color paint and helps to transfer the paint to a surface such as paper. When all moisture evaporates, *Acacia* gum, typically, does not bind the pigment to the paper surface, but is, totally, absorbed by deeper layers.

If little water is used, after evaporation *Acacia* gum functions as a true binder in a paint film, increasing luminosity and helping prevent colors from lightening. Gum Arabic allows more subtle control over washes, because it facilitates the dispersion of the pigment particles. In addition, the gum slows evaporation of water, giving, slightly, longer working time.

The addition of a little gum Arabic to water color pigment and water allows easier lifting of pigment from paper and thus can be a useful tool when lifting out color when painting in water color.

Gum Arabic has a long history as additives to ceramic glazes. It acts as a binder, helping the glaze adhere to the clay before it is fired, thereby minimizing damage by handling during the manufacture of the piece. As a secondary effect, it also acts as a deflocculated, increasing

the fluidity of the glaze mixture but also making it more likely to sediment out into a hard cake if not used for a while.

The gum is normally made up into a solution in hot water (typically 10–25 g/litre), and then added to the glaze solution after any ball milling in concentrations from 0.02% to 3% of gum Arabic to the dry weight of the glaze.(Harman, 1973) On firing, the gum burns out at a low temperature, leaving no residues in the glaze. More recently, particularly in commercial manufacturing, gum Arabic is often replaced by more refined and consistent alternatives, such as CMC.

The historical photography process of gum bichromate photography uses gum Arabic mixed with ammonium or potassium dichromate and pigment to create a coloured photographic emulsion that becomes, relatively, insoluble in water upon exposure to ultraviolet light. In the final print, *Acacia* gum, permanently, binds the pigments onto the paper.

Gum Arabic is also used to protect and etch an image in lithographic processes, both from traditional stones and aluminum plates. In lithography, gum by itself may be used to etch very light tones, such as those made with a number five crayon. Phosphoric, nitric or tannic acid is added in varying concentrations to the *Acacia* gum to etch the darker tones up to dark blacks. The etching process creates a gum adsorbed layer within the matrix that attracts water, ensuring that the oil based ink does not stick to those areas. Gum is also essential to what is

sometimes called paper lithography, printing from an image created by a laser printer or photocopier.

Gum Arabic is also used as a water-soluble binder in fireworks composition.

1.2.7 Calcium element:

Name: Calcium

Symbol: Ca

Atomic Number: 20

Atomic Mass: 40.078 amu

Melting Point: 839.0 °C (1112.15 K, 1542.2 °F)

Boiling Point: 1484.0 °C (1757.15 K, 2703.2 °F)

Number of Protons/Electrons: 20

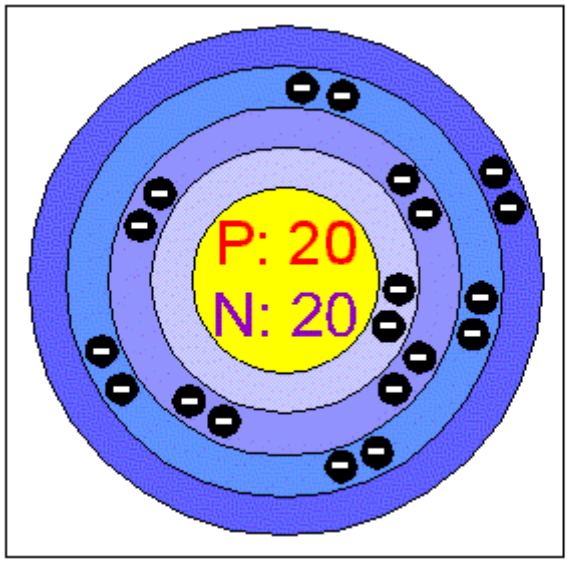
Number of Neutrons: 20

Classification: Alkaline Earth

Crystal Structure: Cubic

Density @ 293 K: 1.55 g/cm^3

Color: Silvery



Number of Energy Levels: 4

First Energy Level: 2
Second Energy Level: 8
Third Energy Level: 8
Fourth Energy Level: 2

Isotope	Half Life
Ca-40	Stable
Ca-41	103000.0 years
Ca-42	Stable
Ca-43	Stable
Ca-44	Stable
Ca-45	162.7 days
Ca-46	Stable
Ca-47	4.5 days
Ca-48	Stable
Ca-49	8.7 minutes

Calcium is essential for many body functions. It helps regulate the heartbeat , sends nerve impulses , helps clot blooding clot, and stimulates hormone secretion.

Calcium also is nerve essential for building denser , stronger bones in early life, and for slowing the rate of bone loss in later life.

Adequate calcium intake is important because the human body cannot produce calcium. The body loses calcium every day through the shedding of skin , nails ,and hair , as well as through urine and feces. This lest calcium must be replaced . when the dite dose not contain enough calcium to perform the necessary activates , calcium is taken from the bonus, where it is stored.

1.2.8 Calcium carbonate :

UPACA name : calcium carbonate .

other names : limestone – calcite – chalk – aragonite – marble – pearl – oyster

chemical formula : Ca CQ3

molar mass:100.0869 g/mol

Appearance: fine white power- chalky taste.

Odor: odorless

Density: 2.711 g/cm^3 (calcite)

2.83 g/cm^3 (aragonite)

Melting point: 1.339 C° (2.442 F, 1.612K) (calcite)

825 C° (1517F ,1.098K) (aragonite)

Boiling point : decomposes

Solubility in water: 0.013gil (25C°)

Solubility product (Ksp) : 3.3×10^{-9}

Solubility in dilute: soluble

Acidity (PKA): 9.0

Refractive index (nD): 1.59

Crystal structure : Trigonal

Space group: 32/m

calcium carbonate: is chemical compound with the formula $CaCO_3$. it is a common substance found in rocks as the minerals calcite and aragonite

(most notably as limestone) and is the main component of pearls and the shells of marine organisms, snails, and egge.

Claim carbonate shares the typical properties of other carbonates not ably

it reacts with acid, releasing carbon dioxide:

$$CaCo_{3(s)} + 2H^{+}_{(aq)} \longrightarrow Ca^{2+}_{(aq)} + Co_{2(g)} + H_2O$$

Calcium carbonate with react with water that is saturated with carbon dioxide to form the soluble calcium bicarbona to

$$CaCo_3 + Co_2 + H_2O \longrightarrow Ca(HCo_3)_2$$

1.2.9 Objective of research:

The objective of this project are:

- To determine the physio chemical properties of *Acacia seyal var sayal* gum accordung to international specifications requirements.
- To decolonrze *Acacia seyal var seyal* gum (talha) using hydrogen peroxide (H_2O_2)
- To remove all cations in *Acacia seyal var seyal* gum (talha) by ion exchange chromatography.
-To prepara of calcium glucouronates from *Acacia seyal var seyal* (talha)

Chapter Two

Materials and Methods

Materials two

Materials and methods

2.1 Materials:

Gum sample - distilled water – hydrogen peroxide 6%W/V - calcium carbonate.

Gum sample:

Aseyal var seyal gum was collected from burm , south Darfur, during season 2013and dented by NoPEc company.

Preparation of crude Gum :

Gum nodules were dried at room temperature, and then cleaned by hand to ensure that they were relatively free from sand dust and bark impurities and kept in labeled container for analysis.

2.2 physicochemical Analytical Methods.

The following analytical methods were emplyedin this study.

2.2.1 Moisture content

A porcelain crucible was dried in an oven at 105c for 30 minute. Cooled in desiccators and weighed (w1) about two grams of the sample were placed in the crucible and weighed accurately (w2) , heated for 5 hours at 105°c , cooled in desiccators and reweighed (w3) .

The loss on drying was calculated as following .

moisture content (%)= $\frac{w2-w3}{w2-w1} \times 100$

Where:

W1: weight of the empty crucible .

W2: weight of crucible + sample be for drying .

W3: weight of crucible + sample after drying.

2.2.2 Ash content:

A porcelain crucible was dried by heating, cooled in a desiccators and weighed (w1) about 2.0 grams of gum sample were accurately weighed in the crucible (w2), ignited at 550° in a heracus electronic muffle furnace until free from carbon, cooled in a dedicator and weighed (w3). The total ash 90 was calculated.

$$\text{Ash (\%)} = \frac{w3-w1}{w2-w1} \times 100$$

W1= weight of the empty crucible.

W2= weight of the empty crucible + sample

W3= weight of crucible + Ash

2.2.3 PH Measurement:

The PH of 1% a queues gum solution using a microprocessor PH meter combined PH or RP electrode , USA was determined.

2.3 Method : dissolving

75g from gum Arabic was dissolved in 500mL distilled water. For three hours, from this sample we take 100ml+ 65m H_2O_2 and slow heating for 20 minute to reduce the color. The residues form the solution 140+ 174 H_2O_2+ heating 70° c and then the color became pale yellow.

2.3.1 Decolourization:

The Resin was activated by adding 12m from H_2SO_4 and then 250 ml / from water was added.

2.3.2 Column separation :

4 ml of Barium chloride was added to detected the precenses of Ba So_4 – when we added 4 ml of Ba So_4 there is no formation of any precipitate . then the solution was added to the column.

2.3.3Neutralization process:

90 g from $CaCO_3$ were added to the solution and the solution was left for 6 days to nuteralize.

After it novelized and the shape become like surly. The sample was dried almost from moisture for one day.

The concentration of calcium (Ca) was determined by Atomic Absorption and the concentration of potassium (K) was determined by flame photometer.

Chapter Three

Result and Discussion

Chapter Three

Result and Discussion

3.1 Physio chemical properties of Aseyal var seyal gum

Table (3.1) shows physicochemical properties of *Aseyal var seyal gum*, the moisture contetnt was found to be (10.14%) it complies with the internation speicfiction not more than (15%)

Total ash content was found to be (3.42%) compare with international Specifications for pharmaceutical and food industry Requirement not more than (4.0%).

The PH of gum solution is found to be (5.53) and PH after ion exchange is found to be (3.80) the Range should be to be within (0-7) that the gum solution is acidic.

Table (3.2) shows cationic composition of *Acacia seyal var seyal gum* .. The calcium content has a highest ratio compared to other elements the other elements like potassium is low.

3.2 Result and determination of calcium

Ca(422.7nm)

Analyst:
File Comment:

Comment:
Flame

Optics Parameters

Element:	Ca
Socket#:	8
Lamp Current Low(mA):	10
Wavelength(nm):	422.7
SlitWidth(nm):	0.5
Lamp Mode:	BGC-D2

Atomizer/Gas Flow Rate Setup Fuel

Gas Flow Rate(L/min): Flame	2.0
Type:	Air-C2H2
Burner Height(mm):	7
Burner Lateral Pos.(pulse):	0
Burner Angle(degree):	0

Measurement Parameters Order:

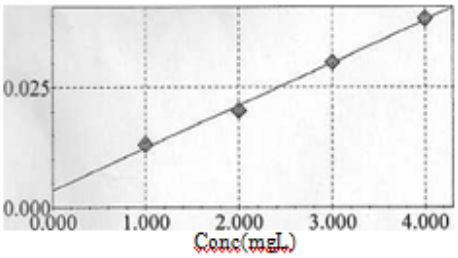
	Num Reps.	Max Reps.	RSD Limit	SD Limit
Blank	1	1	99.90	0.00000
Standard	1	1	99.90	0.00000
Sample	1	1	99.90	0.00000
Reslope	1	1	99.90	0.00000

QA/QC Parameters

	Standard Value	Out of Control Remark

Calibration Cie (C= : 01)

Abs



Cone (mg/L)	Abs
1.0000	0.0131
2.0000	0.0202
3.0000	0.0303
4.0000	0.0393

Abs=0.O0887Conc+O.OO355
r=0.9978

STDl:STD
--0.200-0.100 0.000 0.100 0.400 0.600 0.700

True Value Abs. BG *eft*
1.0000 0.0131 -0.0050 01

STD2:STD
0.000 0.100 0.600 0.700

True Value Abs. BG *Cft*
2.0000 1.0202 -0.0078 01

STD3:STD
...r 0.100 0.100 0.200 0.300 0.400 0.500 0.700 0.800

5.000----------
True Value Abs. BG *Ctt*
3.0000 1.0303 -0.0105 01

STD4:STD
ية200 -0.100 0.000 0.100 0.200 0.400 0.500 0.600 0.700

5.000
True Value Abs. BG *eft*
4.0000 1.0393 -0.0128 01

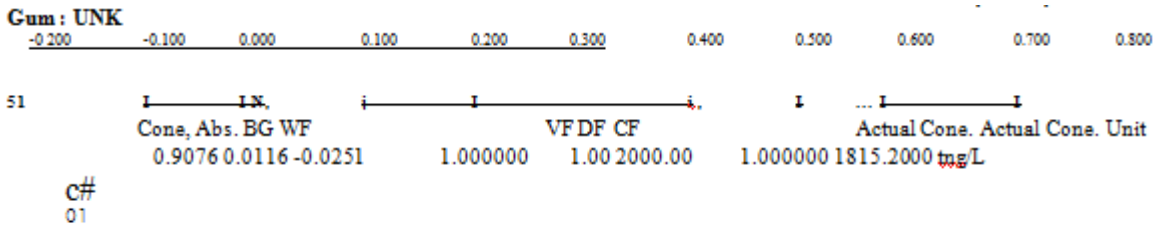


Table 3.1 : physicochemical properties of *Acacia seyal var, seyal gum* sample

Parameter	Test	Results
1.	Moisture content	10.14%
2.	Moisture factor	1.128%
3.	PH (gum solution)	5.53
4.	PH (after ion exchange)	3.80
5.	PH (after neutralization)	11.20
6.	Total Ash	3.42%

Table 3.2: cationic composition of *Acacia seyal var. seyal gum* sample :

Elements	Results(ppm)
Ca	1815
K	0.0793

3. neutralization of *Acacia seyal var. seyal gum*:

The neutralization process increase the ratio of calcium element> (K) than other element in gum sample.

3.3 comparison of PH before and after ion exchange:

Table (3.1) show pH results of sample gum before and after ion exchange using column chromatography the PH values were decrased when the gum solution converted to glucourantes this show that the removed of cation in gum

- the pH after the addition of calcium carbonates was found to be (11.20).

Conclusion :

- Physiochemical properties indicate that the gum used is real *Acaia Seyal var. seyal gum.*
- The neutralization process increase the ratio of calcium element in gum sample > potassium and other elements.
- Addition of calcium carbonate its preparation calcium glucouronates. of gum (talha).

Recommendation:

- It is recommended to use gum Arabic because it have many important in our life:
- Emulsify and keep fatty components evenly distributed.
- *Acacila gum* has been used successfully in a variety of pharmacercial products because of its many functional properties such as binder adhesive and glaze for pharmaceutical tablets.

- It is recommended to use calcium glucourontate product because it medicates osteoarthritis and this product is less toxicity compare with other chemical products.
- The calcium glucourontate want is natural product because the basic content is the gum Arabic and we collected advantages of calcium and gum Arabic (Talha).

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