PROCESSING AND VALIDITY DATE

DETERMINATIO OF INSTANT KARKADE DRINK

(*Hibiscus sabdariffa*)

By

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ABSTRACT

This study was performed to determine the best mixing formula for a hibiscus extract concentrate extracted by water at 50 ˚C for one hour and concentrated by evaporation under pressure to 32 to 40 brix, and dried on sugar powder to make instant hibiscus granules.

Four concentrates were prepared (D: 32, C 36, B 39 and A 40brix) Thirty liters from each concentrate were taken with fifty kg milled sugar powder and the drying process on the powder was carried out using the fluid bed machine then stored fore the required time .

The concentrates were then treated separately with A: gum Arabic, B: anti caking agent CMC (sodium carboxy methyl cellulose), C : gum Arabic and anti caking agent and D: without any additive .

The four treatments were analyzed physiochemical and microbiologically to see the extend of changes during the storage period .

The treatments were stored for one year in an aluminum foil coated with plastic bags to determine the expiry date through testing the physiochemical and microbial changes during the storage period .

Chemical analysis showed significant difference ($P \leq 0.05$) in the four treatments in moisture content ,where D treatment had the lowest moisture content during the storage period whereas C and B treatments were altered at the high moisture content .

the total acidity chemical tests showed significant difference ($P0 \leq 05$) between D treatment and the other three treatments while the later didn't show significant difference in total acidity between treatments within the first and third months.

Significant difference ($P \leq 0.05$) in pH value for the four treatments in the first month were shown .Treatments A and B gave almost similar identical values at ($P \leq 0.05$) 2.56 , 2.59 where C and D values had been 2.46 , 2.50 respectively i.e., there was no significant difference at ($P \leq 0.05$).

During the last month of storage a significant increase occurred for all treatments 2.62 for A and B, also 2.25, 2.47 for C and D respectively .

Physical tests showed insignificant difference ($P \leq 0.05$) for all treatments during storage period but within treatments themselves for A and B the solubility rate was 22 seconds compared with C and D which recorded 27, 29 seconds respectively.

For the bulk density D treatment recorded the highest value during the storage period which was 0.606g/ml . A treatment on the other hand recoded the lowest value of 0.525g/ml. And there was insignificant difference P≤0.05) for each treatment in bulk density during the storage period .

خلاصة الأطروحة

أجريت هذه الدراسة لتحديد أفضل نسبة تركيز لمركز الكركدي المستخلص بواسطة الاستخلاص المائي عند درجة 50 م لمدة ساعة والمركز باستخدام تقنيـة التبخيـر تحت الضغط-80 Kp حتي تركيز32 ـ ـ ـ 40 بركس والمجفف علي طحـين السكر لعمل حبيبات الكركدي المسكرة سريعة الذوبان .

تم اختيار أربعة تركيز هي 32%36% 39%40 % بركس وتم أخذ 30لتر من كل تركيز مع 50كيلو من طحين السكر باستخدام ماكينة تجفيف وتم تحديد الزمن اللازم عولجت المعاملات الأربعة.

A عولج تركيز 40% مع الصمغ العربي

B عولج تركيز 39 % مع CMC مضاد التلبك.

D عولج تركيز 32% بدون أي إضافات .

واختبرت كل من المعاملات الأربعة للاختبارات الفيزوكيميائيـة ومايكروبايلوجيـا تمت تعبئة المعاملات الأربعة في عبوات من شرائح الألمونيوم تم تخـزين الأربعـة منتجات في درجة حرارة الغرفة لمدة 3 أشهر، سته اشهر، تسعة اشهر،وعام لتحديد التغير في الخواص الفيزيوكيميائية و المايكروبايولوجية أثناء فترة التخزين .

أثبتت التحاليل الكيميائية وجود اختلافات معنوي عنـد مـستوي P<0 . 05 فـي المعاملات الأربعة تفاوتا فيما بينها من المحتوي الرطوبي حيث أعطت المعاملة D أقل محتوي من الرطوبة طيلة فترة التخزين بينماC,B أعطت تفاوتاً في المحتـوي الرطوبي.

كذلك أظهرت التحاليل الكيميائية وجود اختلاف معنوي فـي قيمـة(P<0.05) فـي الحموضة الكلية المقدرة لحمض ليمون بين المعاملة D والثلاثة معاملات الأخـرى التي لم تظهر اختلاف معنوي بينها في الشهر الأول والثالث من التخزين .

كذلك أشارت التحاليل الكيميائية إلى وجود اختلاف معنوي عند مـستوى (P<0.50) 2.56 بدون اختلاف معنوي بينما أعطت المعاملات D,C قيمـة 2.50,2.46 علـى التوالي بدون اختلاف معنوي بينها ، بينما تختلف معنويا مـع AB بينمـا أظهـرت الشهر الأخير لفترة التخزين زيادة معنوية (P<0.05) في قيمة PH لكل المعـاملات حيث كانت 2.62, 2.62 لكل من المعاملتان ABعلى التوالي ، 2.47, 2.25 لكل من المعاملتان D,C على التوالي .

أشارت الاختبارات الفيزيائية لعدم وجود اختلاف معنوي لكل المعاملات أثناء فتـرة التخزين ولكن أظهرت اختلافا معنويا فيما بين المعاملات حيث أعطـت المعاملتـان B,A معدل ذوبان في الماء 22 ثانية مقارنة مع المعاملتان D,C حيث سجلت 27، 29 ثانية على التوالي .

في الكثافة النسبية أعطت المعاملة D أعلى قيمة 0.606 جم/مل طيلة فترة التخـزين بينما أعطت المعاملة A اقل قيمة 0.0525جم/مل ، ولم تظهر أي اختلاف معنـوي (P<0.5)في المعاملات طيلة فترة التخزين

CHAPTER ONE

INTRODUCTION

The hibiscus herb (*Hibiscus sabdariffa* L) is considered one of the herbal crops which grow in Sudan at the western states which are characterized by rich mineral sandy soil .

The hibiscus herb is known in the Sudan as (Karkade) and (Angara). The main types grown in the Sudan are the Alrahad and Um Rawaba cultivars.

Karkade is a red flower containing acids , fibers and pigments , which when soaked in water give a dark red extract having a high ratio of acids and considered as a rich source of ascorbic acid (vitamin C), the extract also is rich in calcium and iron .

Sudan is considered as one of the largest countries producing and exporting hibiscus herb to both European and Middle East Market . Most of the herb is exported as raw material for medical and nutritional uses.

Recently many studies were conducted to make use of the herb as a raw material in many nutritional and traditional food processing , these studies where carried out at the food research center and delt mainly with extracting hibiscus via water extraction and then made use of spray drying technique to produce dried hibiscus powder , after

that the powder was further processed by mixing it with sugar powder to make agglomerate ,also jam and gelly were produced from the herb beside many other products.

In this study use was made of technology of water extraction of the hibiscus calyces by automatic extractor then followed by a concentration process which was done making use of the evaporation under vacum at 65 ˚C to increase the total soluble solids of the calyces extract without using high temperature.

The hibiscus concentrate was further processed by spraying on the commercial sugar powder in the fluid bed machine to make instant granulars having almost the same characteristics of the fresh hibiscus calyces .

The study was carried out at ELNASR hibiscus and food processing factory at khartoum north industrial area . the results gave product of good quality which potentially can compete in international market and of good acceptability in local market especially during Ramadan month.

The objectives of this study are :

-To determine the best hibiscus liquid concentrate suitable for production

of fine granules having good characteristics.

-To determine the maximum storage period i.e. validity period of the instant hibiscus dried products .

-Introduce to the world markets a new tropical, processed product out of a Sudanese local raw product (Roselle).

CHAPTER TWO

LITERATURE REVIEW

2.1Hibiscus rosell and it uses

Hibiscus sabdariffa L is a tropical crop . the plant is grown as a fibre crop in many countries however , in the Sudan it is grown for its calyces , the calyces are used for extraction of soluble matter which is used as a beverage .

The crop is grown in African countries where it is considered as a midicinal plant .It is reported to be digestive , emollient and stomachache .

Hibiscus herb is a folk , vever and abscess . The flowers contain anthocyanin and glucoside hibisin , which may have effects on decreasing the viscosity of the blood and reducing the blood pressure . Hibiscus herb calyces is used in food as additive in making marmalade, jelly , ices, sherbets, also the seed contain about 17% oil which is similar in properties to cotton seed oil (Duke , 1983).

2.2Chemical composition of Hibiscus

Reaubourg and Monceaux (1940) pointed out that *Hibiscus sabdariffa* dried fruits of American and Abyssinian origin contained about 15% moisture,the dry matter consisted of 3.5% protein, 10% oil, 63.5% glycoside, 11% cellulose, 12% ash, the glycoside consists of 22% organic acid, 16% reducing Sugars and 25% other nitrogenous substances.

2.2.1Acids content

Organic acids are important constituents of food products, not only as chelating agent for iron and copper but for control of pH and inhibition of enzymes (Sistrunk and Cash,1973).According to Watt and Breyer (1962) the acids constitute about 13% of the whole Karkade Calyces.

The acids present are citric acid and hibiscus acid ($C_6H_6O_7$),hibiscus was first confused with citric acid , because several reactions specially those involving colour changes are the same. Griebel (1939) suggested that it was lactose of hydroxy citric acid with two asymmetrical carbon atoms ,with the formula $C_6H_6O_7$.

Other water soluble acids are malic acid, tartaric acid (trace)while oxalic, garlic, tannic and lactic acids are absent in the aqueous extract (Reubourg and Monceaux, 1940). 3-indolyl acetic acid was also reportedin some Sudanese varieties (Ibrahim *et al* (1971).Organic acids are water- soluble , colourless liquids or relatively law meltingpoint solids. The majority are

non- volatile .Organic acids are classified chemically according to the number of carboxylic acid groups or to other functional groups present. Some of the organic acids shown to be present in aqueous extract of calyces of *Hibiscus sabdariffa* are the followings, mono carboxylic acid (Ascorbic),dicarboxylic acid (Malic), and tricarboxylic acid (cirstic),(Harborne, 1984). Flavor characteristics of Hibiscus were found to be mainly in the acid content.

The presence of citric, malic, olic , lactic , tannic and oxalic acids have all been reported in Hibiscuss (Pritzker and jungunz, 1937) . Hibiscus acid in the calyces was found to be very soluble in water and alcohol and slightly soluble in other solvents , the amount of Hibiscus acid in plant samples analyzed by Grieble (1939) was 13.6% this result stands in agreement with the result obtained by Bachtez (1948) who found 15% in Mexico samples, and 14.6% in Ethiopian samples.

The presence of vitamin C (L. ascorbic acid) in Hibiscus has been confirmed by many analysts among them Mclean(1973) who showed that vitamin C concentration varies from 21 to 89 mg/ 100 g in both frich and dried samples Ibrahim *et al*. (1971) reported ascorbic acid in Roselle water extract with a concentration of 7.12 mg / 100g . Storing dry Hibiscus calyces for more than six months depletes them from vitamin C. (Mario 1959, Addo 1981) .

The total acidity in the dry calyces can be expressed not only as hibiscic or citric , but also as tartaric acid (Schilcher 1976) . Analysis of organic acids in calyces showed their content to range between 8.2 and 12.6 % as reported by Milkowska and Strzelecka (1995) .

2.2.2Amino acids:-

According to the work carried by Ibrahim *et al.*, (1971) the protein content of whole calyces of Sudanese varieties range between 7.05 – 9.49% thirteen amino acids were given by the protein hydrolyzates of the whole and spent calyces, while the protein hydrolyzates from the water extract give only nine amino acids , of the thirteen amino acids six are essential .

The presence of additional amino acids in both whole and spent calyces indicated that these amino acids were present in water insoluble protein factor.

2.2.3 Fats content

It was reported that the nutritive value of Roselle seeds exceed that of linseed or cotton seed cake because of its high fat content (Haarer, 1952). The seeds contain about 17%of oil which is similar in properties to cotton seed oil.

The free fatty acids content of the oil was 170.45 mg KOH/g of oil. The unsaponified matter 1.64% (Rahamma,1979) which yielded B-sitositrol

by column chromatography and crystallization (Hamidi *et al* .1966).The fatty acid composition of the seed was myristic acid trace, palmitic acid 26.38%,stearic acid 4.8 ,oleic acid 32.97% (Rahamma,1979).

2.2.4Carbohydrates content

Hamid et al (1966) detected galacturonic acid and rhamnose in the calyces .

Two other free sugar, glucose and arabinose were also detected in the water extract by Ibrahim *et al* (1971). Brand (1942) detected3.0% sucrose in the calyces who related its presence to the secretory function of the flower.

The pectin content had been shown by Riaz (1969) to increase in both quantity and quality with sepal development during maturation, but it is unlikely to prove commercial source of material in competition with apple and citrus.

The content had been found to be only 3.19% but nevertheless gives the plant its mucilaginous character.

2.2.6 **Ash content**

Analysis of the nonvolatile carbon free residue from Careful Combustion gave about 9% of the calyces on the dry basis. The presence of sodium, potassium, calcium, aluminium, magnesium, and iron was reported by Mcklean (1973).

The presence of manganese was indicated by the intence blue-green color of the ash . Calcium is most copious, followed by phosphorus and iron.

2.2.7 Pigments content

The flower petals of Hibiscus sabdariffa contain hibiscitrin as the main component , gossypitrin is present to small extent . Besides these two , a small amount of new compound named Sabdaritin, has been isolated which on boiling with dilute H_2SO_4 yielded a new hydroxyl flavone called sabdaretin (Suryaparkasa and Seshadri,1943).

The red extractable matter from the calyces had been isolated by Yamamoto and Oshima (1932) in crystalline form .they called it (Hibisin).They pointed out the structure to be cyanidin-3-glucoside (Cy-3-G).They changed it later (1936) to delphinidin- pentoside –glycoside. Watt and Breyer (1962) also mentioned the presence of gossipetin and hibiscin. The pigment of roselle was further examined by Shibata Furukwa (1969) who reported the presence of cyanidine-3-glucoside and

delphinidine -3-sambubioside. A more detailed work carried by Du and Francis (1973) using paper chromatography revealed the presence of more than these two pigments.

Anthocyanins identified were cyanidine-3-glucoside which on complete hydrolysis gave cyanidine and glucose. Cyanidin-3-sambubioside was also present which was the second major anthocyanin. Delphinidin-3-glucoside was identified as a minor component . The most abundant anthocyanin, in Roselle and the pigment responsible for its reddish-violet colour was delphinidin-3- sambubioside which on complete hydrolysis gave delphinidin glucose and xylose .Three other pigments were not identified since they were present in trace amounts .

The total anthocyanin content was calculated as 1.5 mg/100g on dry weight basis expressed in terms of delphinidin-3-glucoside .The work carried by Tamenn (1973) though not elaborated , but still he obtained comperhensive results. He traced the colour fading reaction of the bottled drink of karkade , and he concluded that it was very propably due to hydrolysis which was largely affected by temperature , at 100 ˚C it proceeded 300 times as fast as at 20 ˚C.

These results were in agreement with the work carried by Everett (1953) who stated that the most important factor in changing the kinetics in the degradation of colour in strawberry products was temperature . He

found that the rate of colour deterioration increases in proportion to the logarithm of temperature. At 38 ˚C the half life was 24 hours, at 20 ˚C it was about 1300 hours , while at 4 ˚C it was about (6000-8000)hours.

Tamenn (1973) also observed that contact with iron, copper, zinc, aluminum and tin spoiled the color .Stainless steel had no effect. Light at lower temperatures speeded the reaction somewhat up, abundant contact with air had not influenced the color, it did stimulate precipitate formation. He also noticed that the addition of the ascorbic acid discolored the extract proportional to its dose.

Everett(1953) also pointed out the influence of ascorbic and dehydroascorbic acid on pigment degradation of strawberry products, as being directly proportional to their concentration ,ascorbic acid being more effective and that the reaction was catalyzed by the presence of iron and copper .Iron and copper in themselves were not important contributors to the pigment loss mechanism, except in so far as they hastened the distribution of the ascorbic acid .At 38 ˚C the products in turn attack the pigment in an increasing rate

The more acidic the Karkade solution the more it was stable, increasing pH decreases intensity in color which was not or only partial restored on acidification (Tamenn 73).According to the work carried by

Daravingas and Cain (1968) raspberry anthocyanins were cyanidin -3-diglucoside , cyanidin -3- glucoside and cyanidin-3-,5-rhamonoglucoside-5-glucoside, with cyanidin -3-diglucosides as the main component ,the rate of degradation decreased .The maximum pigment retention was attained at pH 1.8 for strawberry anthocyanins .

Tamenn (1973) also observed that bacteria and moulds as long as they did not influence the pH, were not the cause of the colour fading nor was it an enzymatic reaction. Erlandson and Wrolstad (1972) worked on blanched and unblanched strawberry purely to certain the role of enzyme on anthocyanin degradation. They concluded that enzymatic degradation was negligible. They also concluded that oxygen had little effect on anthocyanin degradation .The pigment degradation rate was always greater in air than in nitrogen, the rate difference being small but relatively constant.

Water availability was essential for anthocyanin break down, rate of degradation increased as relative humidity increased . The rate was also accelerated by sugars (glucose, fructose , sucrose, sorbitol , gluconic acid, glucuronic acid, maltose and lactose) . Fructose and glucuronic acid were higher in their effect than all the others. The presence of amino acids and sugar together produced a more accelerated rate (Tinsely and Bockian , 1960).

In this respect Labuza et al.(1970)reported that even at low water activities (as low as 0.6-0.7)sucrose hydrolysis could occur giving rise to reducing sugars which had a potential for browing and other reactions .

2.3Processing of hibiscus extract

The work on spray drying of karakade extract was initiated in Sudan Food Research Centre in 1968.The work was mainly concerned with extraction, Spray drying, and powder quality, (Jackson, 1968).

2.3.1**Extraction:-**

The extracting solvent of Karakade calyces is water .Preliminary trials carried out on methods of extraction, had shown that extraction with hot or boiling water produced cloudy extract . Part of the cloud precipitate on standing and apparently contained pectin and gum. Extraction with water at ambient temperature produced a clear sparkling extract .If however, old extractor was prolonged to 24 hours a cloudy extract again resulted (Jackson and Bashir, 1968a).

The quality of water used for making extracts which were to be spray dried was important. Khartoum mains water,when spray dried would leave a considerable deposit of slightly alkaline salts. These salts affect the colour of karkade extract which presumably resulted in colour degradation , they also influence the pH of the spray dried powder .For these reasons only destilled water was being used in extraction trails

(Jackson & Bashir ,1968).

It was observed that there was an increase in percentage extraction as the ratio of water to karakade was increased .Extraction of about 60%of the weight of the raw material was obtained when 100 parts of water to one part of karakade was used . The ratio of 4:1 extraction gave only about 15% also ,a decrease in particle size increased persentage extraction and required less water (Bashir,1969).

It was desirable that the extract for spray drying shoud be as concentrated as possible,since the cost of evaporating water in spray drier was high .On the other hand production of a highly concentrated extract might mean that the raw material had to be discarded before it was completely exhausted .on the other hand when a dilute extract of 4.5% had to be concentrated e.g to 32% it had to undergo a long heat treatment at 60 ˚C.

Karakade anthocyanins as had been previously mentioned were very tempreture sensitive and that heat treatment had an adverse effect .

Water is a precious commodity in Babanousa where karakade powder is produced in commercial scale. The temperature of the cooling water to condense the vapour at 60 ˚C was about 35 ˚C meaning that for this condensation an enoromous quantity of water was needed .

(Tamenn,1973). Small scale continuous countercurrent series extraction apparatus was developed.

Tamenn,(1973) also deviced an apparatus of interconnected compartments,which gave a constant flow of 32% concentration, but still improvements on the extraction method are possible.

2.3.2Concentration of Hibiscus calyces

The concentration of liquid foods permits economies in packing since the volume and wight of the food are reduced as well as storing and transporting space . The concentration permits the development of new product , it also permits the economic utilization of perishable crop during peak harvesting periods ,contributing to the stabilization of production and of consumer prices (Heide and Casten,1961).

The concentrated form of some foods have become desirable components of the diet in their own right . The concentration of food can be also concidred as a form of preservation .

2.3.2.1Vacuum Evaporation

For the evaporation of foods which are affected by high temperatures , it may be necessary to reduce the temperature of boiling by operating under reduced pressure . The relationship between vapour pressure and boiling temperature for water is as follows ,when the vapour pressure of

the liquid reaches the pressure of its surroundings, the liquid boils . The reduced pressure required to boil the liquid at lower temperature are obtained by mechanical , or steam jet ejector , vacuum pumps .

Mechanical vacuum pumps are generally cheaper in running costs but more expensive in terms of capital than are steam jet ejector(Earle 1982).

2.3.3Spray drying

Water extracts of karakade had been used for producing spray dried karakade powder The yield of powder was roughly about 25% of the original weight of the dry calyces .The powder was of good colour and when reconstituted was close to the colour and flavour of the water extract of the calyces(Jackson & Bashir, 1968).

Spray drying conditions are: water at 240-250 ˚C inlet temperature, 90 – 100 ˚C outlet temperature and 1.5 k/g cm compressed air pressure (Bashir 1969).

The optimum extract concentration as far as maximum yield was concerned has not been specified yet, although preliminary work suggested 10 -14% to be optimum because above this concentration much powder was deposited on the walls of the drier which cause a drop in the yield .

These results however ,have not been certified yet (kareem,1975).

Tammeen(1973) suggested that spray drying of highly concentrated extracts could be improved by specially plating the walls of the drier.

2.3.4**Quality of the powder**:-

Spray dried powder made from hot water extracts were not completely soluble and had dull colors (Jackson and Bashir,1968b). Cold soaking water produced powder of good colour and better reconstitution properties (Jackson and Bashir, 1968a). spray drying of highly concentrated extract produced fine powder not easy to reconstitute (Bashir, 1969).

Storage in very tight containers lined with polyethylene bags in a cool place (68 ˚F) lengthened the shelf life of the powder (Bashir, 1969).According to Tamenn (1973) the gauge of the polyethylene bags had to be increased as the package size was decreased.

2.4Some processes for producing granular foods:-

Agglomeration , instantiation and spray drying are often linked together in the food industry, either as simultaneous processes or as consecutive manufacturing steps. Agglomeration is the most important means of instantisation.

In formulating products to enhance their instant properties, some

products require additives either because they are otherwise difficult to agglomerate, or because agglomeration alone was not sufficient to achieve instant properties. Surfactants are often applied, whole milk powder was an important example, but are often combined with other additives or treatments. Sugar is common means of enhancing the instant properties of many beverage products. With proteinaceous powder such as sodium caseinate, addition of sugar is combined with application of surfactants(Jensen,1975).

Many processes of agglomerating foodstuffs were deviced. One process

Was described by Mohr and Nenninger(1969) whereby flour, semolina, starch, protein substances etc… were mixed with a small amount of sucrose (e.g. at ratio 9:1) and the mixture was heated approximately to 90 ˚C by high frequency energy so that the surface of the sucrose particles was liquified and the semolina particle ,ect.. collected around them, thus forming agglomerates when cooled .

Agglomerates of sugar powder could be obtained by simple method. The powder was wetted to a moisture contents less than 20% and was subjected to intense mechanical agitation to form agglomerates which upon drying had a relatively high bulk density(Gyde and Mendl, 1968).

2.5Microbial spoilage of concentrated juice

It is generally accepted that sugar inhibits the growth of mold and bacteria owing to the high osmotic pressure of its solution in high concentration .Abdelrahman(2000).

Fermentation is brought about in sugar products by yeasts and it is not always expected that live yeast can exit in syrup up to a concentration of 80% of sugar (smith and morris , 1952)

Mikki *et al* .(1977)pointed out that the unusual nature of date syrup content make it susceptible to attack only by a limited number of microorganisms known as sugar tolerant yeast , some times called osmophiles . Such yeasts, are capable of growing in concentrates, altering the taste and flavor of the product and making it finally unsuitable for human consumption. Growth of dormant yeast may also be accelerated in diluted syrups . The phenomenon of dilution may be attributed to the fact that during the warm cycle , moisture from syrup distills into the head space of the container , then condenses as free droplets during the cold cycle , eventually collect on the syrup and finally dilute it .The other factors like lack of appropriate plant sanitation measure in date syrup plant and faster multiplication of contaminating organisms due to high temperature prevailing for a long time , may also be involved in the spoilage of date syrup .

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials

3 1.1 Source of herb :-

Hibiscus calyces *Hibiscus sabdariva* **Elrahad** variety ,was purchased from **Elnasr** Company ,and were ground and sieved ina sieve mesh No 16 nm .

3 .1.2 Chemicals and reagents :-

Chemicals and reagents used in this study were of analytical grade .

3.1.3 Additives

Sucrose powder was prepared by milling sucrose of commercial grade (kenana company) in electrical mill . Other additives were CMC sodium carboxy methyl cellulose and gum arabic powder (hashab).

3 .1. 4 Preparation of sample

3 .1 .4.1 Extraction

Hibiscus extracted liquid was prepared from the calyces by soaking the calyces in water at (1 : 0.144) water : calyces .

for three hours at 50 ˚C to produce hibiscus extract at concentration of 7% total soluble solids using soft water which was softened by water softener instrument and sterilized by ozone and UV. Water was tested for hardness, conductivity, pH, turbidity , and total count of bacteria .

3 .1. 4.2 Concentration

Hibiscus extract (7%total soluble solids)was concentrated to 32% ,36% , 39% and 40% , total soluble solids in the forced circulation vacuum evaporation concentrator at pressure -85 bar and steam temperature 58˚C .

` The hibiscus concentrated liquids were tested to determine brix (concentration percentage) ,ascorbic acid content , mineral content (Calcium , Magnesium . Iron), colour , total count of microorganisms (bacteria and yeast)

3 .1. 4.3 Granulation

The hibiscus concentrated liquid was sprayed with sugar powder (commercial grade sugar) . Sugars were milled using an electrical mill to powder . Sugar powder and hibiscus concentrated liquid were used to make Granular in spray drying machine (fluidized bed),

Feeding rate ---------- 40ml \min

Inlet temperature---------- 58 ˚C

Out let temperature---------- 50 ˚C

Four different concentrated liquids (A , B , C ,and D) were used

A 40 brix with gum arabic .

B 39 brix with anti caking agent CMC .

C 36 brix with gum arabic and CMC.

D 32 brix without any additives.

In each batch 30 liters of concentrated hibiscus liquid was sprayed on 50 kg of sugar powder .

The four types of granulas produced were chemically analyzed for moisture content ,total acidity , ascorbic acid , pH value , colour , ash content . protein content , mineral content (calcium , magnesium , iron) , and microorganisms (total count of yeast) .

All types of samples A ,B ,C , D treatments were stored for three months, six months , nine months and twelve months , and then the following tests were carried out:-

The moisture content, bulk density, particular size, solubility rate, pH value , total acidity ,colour and microorganisms growth in

order to determine the characteristics changes and validity period .

3.1.5 Storage of samples

Hibiscus instant (four treatments A , B , C , D) were packed in aluminum foil coated with plastic pack at room temperature (34±4 ˚C) , and stored for one year, every three months a sample was taken and tested to determine changes in physical and chemical characteristics .

3 .2 Proximate analysis:-

3 .2.1 Moisture content

Moisture content was determined by the A O A C method (1975) . A 2.00 grams of sample were dried in an air oven at 103± 2 ˚C for 24 hours .

$$\%\text{Moisture} = \frac{\text{weight loss (g) X 100}}{\text{weight of sample taken (g)}}$$

3 .2.2 Protein content

Nitrogen content was determined by micro - kjeldahl

technique following the A O A C method (1975) . A 0.20 grams of sample was weighed accurately into kjeldahl flask , 0.4 grams of catalyst mixture and 3.5 ml of concentrated sulphuric acid were added .

The flask was placed in the digestion equipment for 2 hours .

The digested sample was then placed in the distillation apparatus ,, 20 ml of 40% NaOH were added and the ammonia evolved was received in 8 ml of 2% boric acid solution , the traped ammonia was titrated against 0.02 M of HCl using universal indicator (Methyl red + bomocresol green) .

$$\%N = \frac{\text{volume of HCl X 0.02 (N HCl) X 14X 100}}{\text{sample weight X 1000}}$$

%Proteine = %N X 6.25

3 .2.3 Ash content

Total ash content was determined according to the AOAC method (1975) . A 2.0 grams of sample were ignited at 500 ˚C in a muffle furnace for 24 hours

$$\%Ash = \frac{\text{weight of ash X 100}}{\text{weight of sample}}$$

3 .2.4 Crude fiber

Crude fiber was determined according to A O A C method (1984) using the fibertic system , 1010 heat extractor . About two grams of defatted sample were weighed . One hundred and fifty ml of the H_2SO_4 (conc. 7.3 ml/1) were added and then heated to boiling . The mixture was boiled for 30 minutes and then filtered . The residue was washed three times with hot water . Then 150 ml of pre-heated KOH (12.8g /l) were added and then heated to boiling ,Then the system was boiled for 30 minutes and then filtered .The residue was washed three times with hot water. It was dried under suction and then in an oven at 105 ˚C overnight and then weighed (W1) .

The residue was ashed in a muffle furnace at 550 ˚C for three hours till a light grey ash was formed and then weighed (W2)

$$\%\text{Crude fiber} = \frac{(W1 - W2) \times 100}{\text{sample weight}}$$

Where :-

W1 : weight of sample before ignition.

W2 : weight of sample after ignition.

3 .2.5 Mineral matter content

Three minerals , namely iron , calcium and magnesium in hibiscus herb ,concentrated liquid and instant were estimated according to the analytical method for Atomic Absorption Spectrometry (Perkin Elmer , 1994) .

The minerals were determined by using an atomic absorption spectrophotometer , (Perkin Elmer Company , Model No . 311) ,at Environment al Research Institute Khartoum .

The ash was dissolved in a five ml HCl (20 %) , the solution was warmed to dissolve residue , filtered through an acid washed filter paper , then the volume of the solution was made up to 50 ml with distilled water , and taken for determination of each mineral .

For determination of Calcium (Ca), Magnesium (Mg), 1 ml of 1% lanthanum chloride was added to the final

dilution .

$$\text{mg mineral /100g sample} = \frac{\text{mg /1 X volume used X 100}}{\text{1000 X weight of sample}}$$

3 .2.6 Total oil content

Total oil was determined according to A O A C method (1975) . A 2.0 grams of sample were extracted with hexane for 8 hours in soxhlet apparatus.

$$\% \text{ Oil} = \frac{\text{weight of extracted oil X100}}{\text{wt of sample}}$$

3 .2. Total carbohydrate

The total carbohydrate was determined by difference : 100 – (crude protein % + crude f ibre %+ crude oil % + total ash % + moisture %)

Moreover , calyces were analysed to determine the following .

3 .3.1 pH Determination

pH of 10 % solution was determined using a glass electrode Fisher pH meter . The pH meter was calibrated with a buffer solution at pH 4.0 (Ruck ,1963) .

The buffer was prepared from potassium hydrogen

phthalate according to A O A C by dissolving 5.12 grams in 500 ml distilled water .

3 .3.2 Total acids

Total acids were determined by titrating 100 ml of 10% solution against 0.1 N NaOH to pH 8.1. Total acids were expressed as citric acid according to the equation :-

$$\%\text{Total acidity} = \frac{\text{1/10X eq .wt of citric acid X normality of NaOH X titration}}{\text{wt . of sample}}$$

Equivalent wt .of citric acid = 70.0 (Ruck 1963).

3 .3.3 Ascorbic acid determination

Ascorbic acid was determined according to the method described by Evered (1959) for highly colored solutions especially that containing red pigments .

Reagents

N .bromo succinaimide solution :A stock solution was prepared by dissolving 200 mg of reagent in warm water , cooling and diluted to 100 ml , this solution was staple for a few days at 4 ˚C .

Just before using the stock solution was diluted (1:9) with distilled water .

One ml of this solution is equivalent to 0.20 mg of ascorbic acid

- Acetic acid : Glacial analytical reagent grade .
- Potassium iodide solution : 4% w\v . aqueous form
- iodide –free potassium iodide .
- Diethyl ether : (Peroxide free) .
- Standard ascorbic acid solution

0.2 mg \ml freshly prepared by dissolving 50 mg of analytical grade ascorbic acid in 1% acetic acid and completing the volume to 250 ml .

The sample was diluted with 1% aqueous acetic acid until it contains 0.4 - 0.1 mg of ascorbic acid \5 ml of solution.

Five ml of the diluted sample were transferred at a 6 inch x 1 inch test tube .

One ml of glacial acetic acid was added ,mixed ; 5 ml of potassium iodide solution were added and mixed again .

Three ml of diethyl ether were added and the mixture was titrated againts N .bromo succinimide solution (added

from 1 ml semi micro burette) .

The test tube was shaken vigorously after each Addition of titrant , and the organic layer was allowed to seperate . The end point was indicated by the first appearance of the brown color of the liberated iodine in the upper ether layer , comparison against an untitrated mixure permitted easy establishment of the end point . A blank was titrated in which 5 ml of potasum iodide plus a volume of water equivalent to the titre replced the diluted sample .

The volume of N-Bromo succinimide solution necessary to import a definate brown colour to the ether layer was determined .

The N-Bromo succinimide was standardized by titrating against 5 ml aliquots of ascorbic acid solution .The equivalent of ascorbic acid to 1 ml of N-Bromo succinimide was obtained.

For eash sample , ascorbic acid content was calculated in mg/100 g of sample as follows

$$\text{Vitamin C mg\100g} = \frac{\text{titre volume X eq. N-bromo sccinimide X100}}{\text{wt of sample in g}}$$

3.3.4 Optical density

According to Alshosh (1997) , a solution containing one g of hibisus instant per 100ml distilled water was used. The optical density which was used to indicate the colour intensity of the extract was measured at half hourly intervals for four hours .An EEL colorimeter adjusted at wave length 535 mu hand .The solution was kept continuously stirred by use of magnetic stirrer .Extraction was allowed to take place at room temperature (34±4 ˚C) .

3 .3.5 Bulk density

A weighed sample 20 g was transferred to a graduated 100 ml measuring cylinder and mounted on a screen vibrator . The bulk density after vibrating for 5 minutes was obtained by measuring the volume occupied in the cylinder and was expressed as g /ml (Kareem , 1973) .

3 .3.6 Solubility rate

This was determined as follows : 10 grams of the sample were added to 250 ml of distilled water at 20 ˚C in 400 ml beaker . The mixture was immediately stirred using a mechanical stirrer at 1000 rpm to assure systemic stirring and counting the time in seconds for the material to dissolve completely .

3 .3.7 Organoleptic Test

Organoleptic test was accessed for duplicate sample by ten judges from the department of Food Science and Technology U of K , for colour , flavor and taste . Samples were scored on a scale of ten points , higher marks indicating better quality .

3.3.8 Statistical analysis

Data obtained for the hibiscus herb and four treatments of hibiscus instant drink were analyzed statistically by using a randamized complete block design (RCBD)according to the method of (39) Gomez and Gomez (1981).

The least significant differance (LSD) at $P \leq 0.05$ was Calculated .

CAPTER FOUR

RESULTS AND DISCUSSION

4.1 Chemical composition of hibiscus calyces and instant drink

4.1.1 Moisture content

As shown in Table 1 the moisture content of the dry hibiscus calyces was found to Be 4.30% , Alshoosh (1987)reported 5.76% to 6.95% for season 93/94 , the result reported by Ibrahim et al (1971) was not in agreement with the result obtained in this study, this may be refered to storage conditions .The hibiscus instant reported 1.30% this result was lower than the result reported by Ismail (1981) for spray dried hibiscus and aglomerate made from spray dried hibiscus mixed with sugar powder .

4.1.2 Protein content

Table 1 shows the protein content of the dry hibiscus calyces to be 7.17% while instant drink reported 1.41% there is significant difference ($p \leq 0.05$) the result of dry hibiscus calyces fall within the range of 8.70% to 13.57% reported by Alshoosh (1987) for six cultivars of karkade from season 93/94,94/95 . Ismail et al (1981) reported a higher value of protein 9.44% for dry calyces , which is in agreement with the protein values reported by Ibrahim et al (1971).

4.1.3 Crude fiber content

Table 1 shows the crude fiber content of hibiscus calyces and hibiscus instant drink . A significant difference (p,0.05) was observed .The fiber content of dry calyces was 15.72% ,while instant drink has no crude fiber .The result reported for calyces was similar with the value of 15.22 reported by Mcklean (1973) for dried calyces of somalian gibiscus , A slightly lower value of 14.60 was reported by Ismail (1980) for dried calyces .Whereas nil % of crude fiber for agglomerate granulated made from sugar powder and spry dried karkade was reported by Ismail (1980) .

4. 1 .4 Total carbohydrate content

The total carbohydrate of Hibiscus calyces and Hibiscus instant drink are shown in Table 1 . A significant difference ($P \leq 0.05$) was observed in total carbohydrate .(59.31%) recorded for crushed calyces, this result is in agreement with the data reported by Alshoosh (1987),and Mclean (1973) for hibiscus calyces .Hibiscus instant drink reported a higher value of 82.0% , this increase in value was due to adding of sugar powder during the production of instant drink.

Table 1 Chemical composition of Hibiscus calyces and instant

Hibiscus types	Moisture %	Protein %	Fibre %	pH	Carbohydrate %	Ash %	Acidity%
Calyces	4.30 a	7.17 a	15.72 a	2.49 a	59.30 b	8.30 a	18.5 a
Instant	1.30 b	1.41 b	0.00 b	2.53 a	82.00 a	2.00 b	4.6 b
L S D	0.148	0.011	0.014	0.132	0.353	0.01	0.081

L S D lest significant difference

4 .1 .5 Ash content

Table 1 shows the ash content of hibiscus crushed calyces and hibiscus instant drink . A significant difference ($P \leq 0.05$)in ash content among the two types was observed. Hibiscus crushed calyces geve 8.3% ,this result was similar to the results range reported by Alshoosh (1987)also the results are in agreement with the data reported by Ibrahim (1971) . 2.0% was reported for hibiscus instant drink, this value was lower than any reported results .

4 . 1. 6 Total acidity (as citric acid)

Table 1 shows the total acidity of hibiscus crushed calyces and hibiscus instant drink , the results given in Table 1 shows significant difference ($P \leq 0.05$) in the total acidity , 18.5% was reported for crushed calyces , this value fall in the range reported by Alsoosh (1987) , a lower value (4.6%) was reported for hibiscus instant , the result reported by Ismail (1981) for spray dried powder gave high acidity content of 34.87%.

4 .1 .7 pH value

Table 1 shows the pH value of hibiscus crushed calyces and hibiscus instant drink , insignificant difference ($p \leq 0.05$) was observed between the values of 2.53 and 2.49 reported for hibiscus instant drink and hibiscus crushed calyces . These results were obtained for samples analyzed in Sudan .

4.2 Water tests

Table 2 shows the characteristics of water used to extract hibiscus calyces before and after purification (tap and soft water) .

4.2.1 Hardness

Table 2 shows significant difference ($p \leq 0.05$)obetween the tap water and soft water ,RW recorded (64 ppm) this result was higher than the result recorded for soft water (Zero ppm),this indicated the effectivness of the purification unit instrument .

4 . 2 .2 PH value

As shown in table (2)there was significant difference ($p \leq 0.05$)between tap and soft water , as tap water had pH value 6.49 and soft water recorded 7.1

4.2. 3 Conductivity

Table 2 shows significant difference ($p \leq 0.05$)in the conductivity , soft water recorded 0.22 this value was higher than the value recorded for tap water 0.144 .

4.2.4 Total count of bacteria

Table 2 shows significant difference ($p \leq 0.05$) in total count of bacteria TCB 71 C/ml recorded by tap water , while soft water recorded zero c/ml .

Table (2)Water test

Water types	Hardness	pH	Conductivity	T C P	Coliform Ba
Raw water	64.0ppm a	6.49 b	0.145 b	71 c/ml a	Nill a
Soft water	0.00ppm b	7.07 a	0.220 a	Nill b	Nill a
S D	0.408	0.047	0.00071	1.079	0.000

T C B : Total cont of bacteria .

S D : Standerd Deviation .

4.2.5 Coliform bacteria

As shown in table 2 there was insignificant difference ($p \leq 0.05$) in coliforin bacteria content for tap and soft water as both recorded zero c/cm.

4.3 Mineral and ascorbic acid content

The mineral content of hibiscus crashed calyces and hibiscus drink are shown in table 3 .

4.3.1 Iron content

Table 3 shows that the iron content of hibiscus crashed calyces and hibiscus instant drink detected significant difference ($p \leq 0.05$) between the two types of hibiscus. The iron content of crash herb calyces was (14.9 mg/100g) and that of instant was(28.5 mg/100g) these results are slightly lower than those reported by Al Alshoosh (1987) 147.3 mg/100g .

4.3.2 Magnesium content

Table 3 shows that the magnesium content of hibiscus crushed calyces and Hibiscus instant drink were 0.25%-0.23% respectively .These results were in fair agreement with those reported by Alshoosh (1987) ,which were 0.24% - 00.25% for Sudanese hibiscus .The variation in values of magnesium content may be attributed to the processing treatment and adding of sugar

Table 2 Mineral content and ascorbic acid of Hibiscus calyces and instant

Types of hibiscus	Iron (mg/100g)	Magnesium %	Calcium %	Ascorbic acid mg/100g
Ka	14.83 b	0.25 a	1.23 a	94.1 a
Kb	28.50 a	0.23 b	0.65 b	42.1 b
S D	0.08	0.0057	0.08	0.10

Legend

Ka : hibiscus calyces .

Kb : hibiscus instant drink .

S D : Standard Deviation.

4.3.3 Calcium content

Table 3 shows the calcium content of hibiscus crashed calyces and hibiscus instant drink.

The results showed significant difference ($p \leq 0.05$) between the two types of Hibiscus 1.23% and 0.65% respectively. Alshoosh et al (1987) reported that the calcium content of karkade calyces for season 94/ 95 . 95 / 96 ranged from 0.53 to3.35 and the mean value was 1.94% .

4.3.4 Ascorbic acid (vitamin C) content

Table 3 show the vitamin C content of hibiscus crushed calyces and hibiscus Instant drink. Vitamin C content of the two hibiscus types was 94.1mg/100g and 42.1 mg/100g respectively this result detected significant decreasing trend ($p \leq 0.05$) from hibiscus crashed calyces to instant (heat decreased the ascorbic acid content). Results similar to those where reported by Alshoosh *et al* (1987) . Presnt results were higher than those obtained by Reaubourg and Monceaux (1940)and Ibrahim et al (1971). Ismail (1980)reported a lower value of ascorbic acid 16.5mg/100g for spray dried hibiscus .

4.4.1Changes in moisture content

Table 4 shows changes in moisture content of the four treatments of hibiscus instant drink (A ,B , C, D) packed in aluminum foil coated with plastic ,and stored at ambient temperature ($34 \pm 4^{\circ}C$). A significant difference ($p \leq 0.05$) in moisture

Table 4 Changes in Moisture content of Hibiscus instant treatments stored at ambient temperature (34±4˚C)

Instant treatments	First month	Third month	Sixth month	Ninth month	Twelfth month
A	1.70 b	1.70 b	1.66 a	1.25 b	1.70 c
B	2.50 a	2.25 a	1.20 c	1.12 b	1.75 b
C	1.71 b	1.50 c	1.60 b	1.70 a	2.00 a
D	1.24 c	1.21 d	1.22 d	1.25 b	1.50 d
L S D	0.029	0.013	0.036	0.0163	0.0163

Legend

A : liquid concentrate 40% treated with gum arabic .

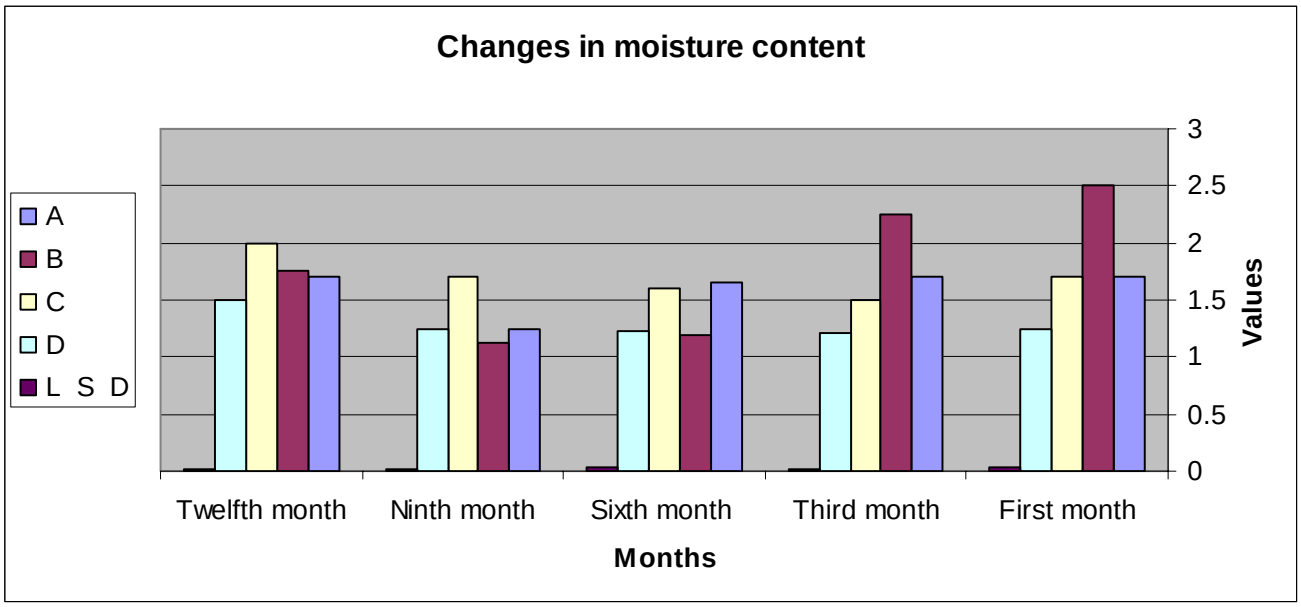
B :liquid concentrate 39% treated with CMC as anti caking agent .

C :liquid concentrate 36% treated with CMC and gum arabic

D :liquid concentrate 32%without any additives .

L S D : least significant difference .

Figer (1) Changes in Moisture content of Hibiscus instant treatments stored at ambient temperature (34±4˚C)



content between the four treatments was observed .B treatment recorded the highest moisture content of 2.50% and the lowest value was recorded for D treatment 1.25% . similar result in agglomerated (sugar and hibiscus powder) was recorded by Ismail et al (1981).

Table 4 showed insignificant changes ($p \leq 0.05$) in moisture content of D treatment during the 3d , 6th and 9th months of storage period ,the significant increase($p \leq 0.05$) reported for D treatment (1.5%)occurred in the 12^{th} month , this result was the lowest value at the end of storage period of all instant treatments . Although A and C have the same moisture content value at the first month of storage , they differ in moisture content at the twelfths month , B treatment gave a high value of moisture content in the beginning of storage period and had significant decrease ($p \leq 0.05$) at the end of storage period .

4 .4.2 Changes in pH value

Table 5 shows the changes in pH values of the four treatments of hibiscus instant drink (A , B , C , D) packed in aluminum foil coated with plastic , at ambient temperature ($34 \pm 4^{\circ}C$) . A significant increase ($p \leq 0.05$) in pH value of treatments during storage was observed for A and B treatments . A significant decrease ($p \leq 0.05$)in pH value was obtained for C and D treatments . The pH values ranged between 2.46 to 2.56 for the four treatments in the first month of storage .

Table 5 Changes in pH values of Hibiscus instant treatments stored at ambient temperature (34±4˚C)

Instant treatment	First month	Third month	Sixth month	Ninth month	Twelfth month
A	2.55 a	2.62 a	2.63 a	2.63 a	2.62 a
B	2.56 a	2.55 b	2.58 b	2.62 a	2.63 a
C	2.46 b	2.50 b	2.46 c	2.50 b	2.26 c
D	2.50 b	2.46 b	2.48 c	2.48 c	2.47 b
L S D	0.043	0.059	0.024	0.021	0.0206

Legend:-

A : Liquid concentrate 40% treated with gum arabic .

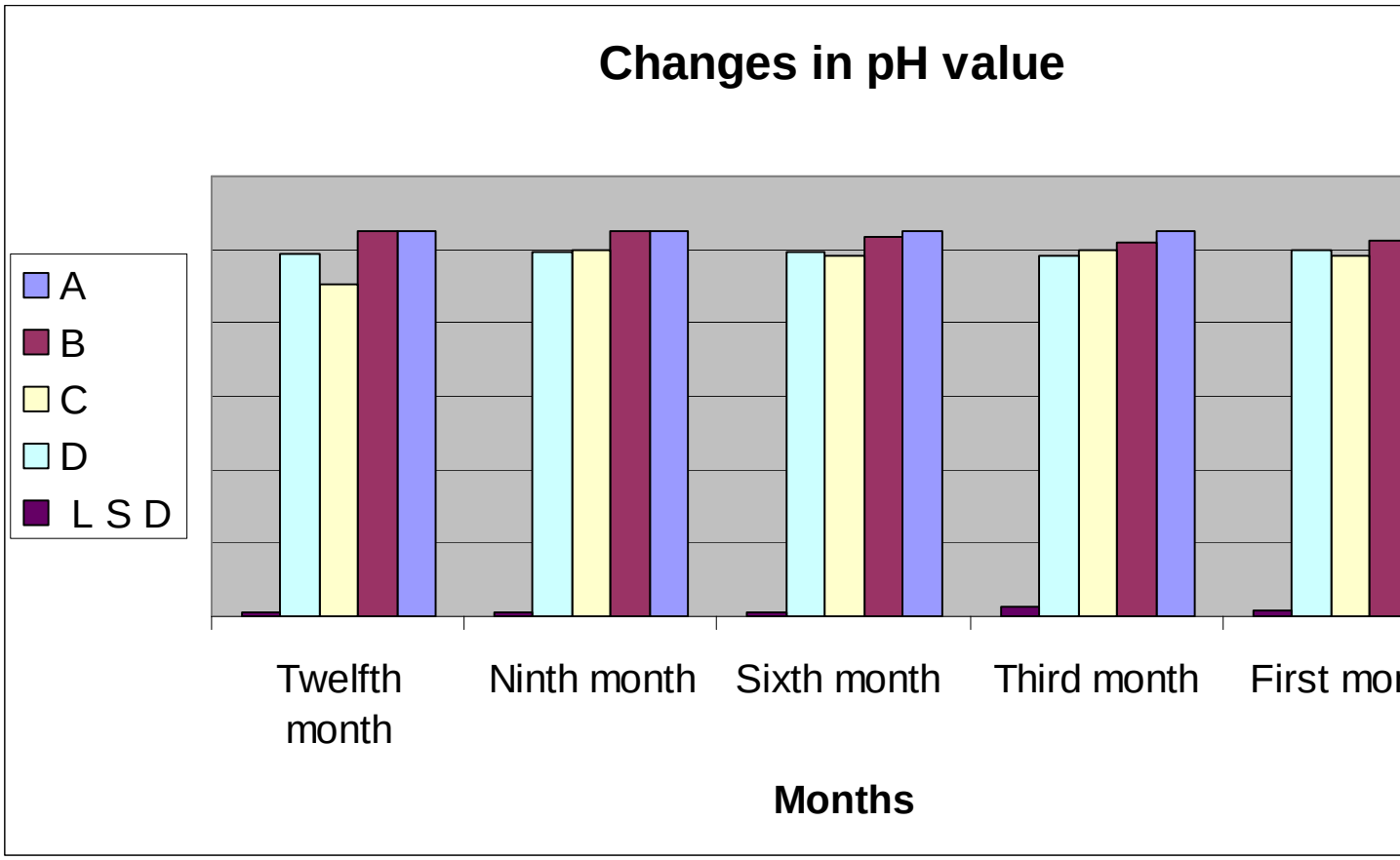
B : Liquid concentrate 39% treated with CMC as anti cacking agent .

C : Liquid concentrate 36 % treated with gum arabic and CMC .

D : liquid concentrate 32 % without any additives .

L S D : least significant difference.

Figer (2) Changes in pH values of Hibiscus instant treatments stored at ambient temperature (34±4˚C)



These results agreed with values obtained by Ismail (1980) which were in the range of 2.63 to 2.70 for agglomerated karkade powder and sugar powder. High value of pH was reported for A and B treatments 2.63 and2.62 at the ninth month of storage period the PH values were also high at the twelfth month of storage . C treatment had the lowest pH value in the beginning and at the end of storage period.

D treatment had nearly a same value at the first month and the twelfth month of Storage .

4.4.3Changes in Total acidity (as citric acid)

Table 6 shows the changes in total acidity of the four treatments of hibiscus instant drink (A , B . C , D) packed in aluminum foil coated with plastic ,at ambient temperature ($34\pm4^{\circ}C$). A significant difference ($P\leq0.05$) in total acidity between the four treatments was observed . C treatment recorded the highest acidity value 4.4% in the beginning but it recorded a significant decreases in the end of the storage period , D treatment recorded the lowest value in the first and twelfth month (insignificant difference ($P\leq0.05$) . B treatment recorded insignificant difference in the first, third , and sixth months of storage period , but starting from the ninth month as shown in table 7 a significant decrease ($P\leq0.05$) for B treatment was observed.

Table 6 Changes in Total acidity of Hibiscus instant treatments stored at ambient temperature (34±4˚C).

Instant treatment	First month	Third month	Sixth month	Ninth month	Twelfth month
A	4.30 b	4.20 c	4.20 c	4.05	4.16
B	4.30 b	4.35 b	4.35 b	4.05	3.85
C	4.42 a	4.10 d	4.05 d	3.93	3.85
D	3.98 d	3.98 d	3.98 d	3.85	3.80
L S D	1.106	0.0556	0.035	0.0249	0.0216

Legend

A : Liquid concentrate 40 % treated with gum arabic .

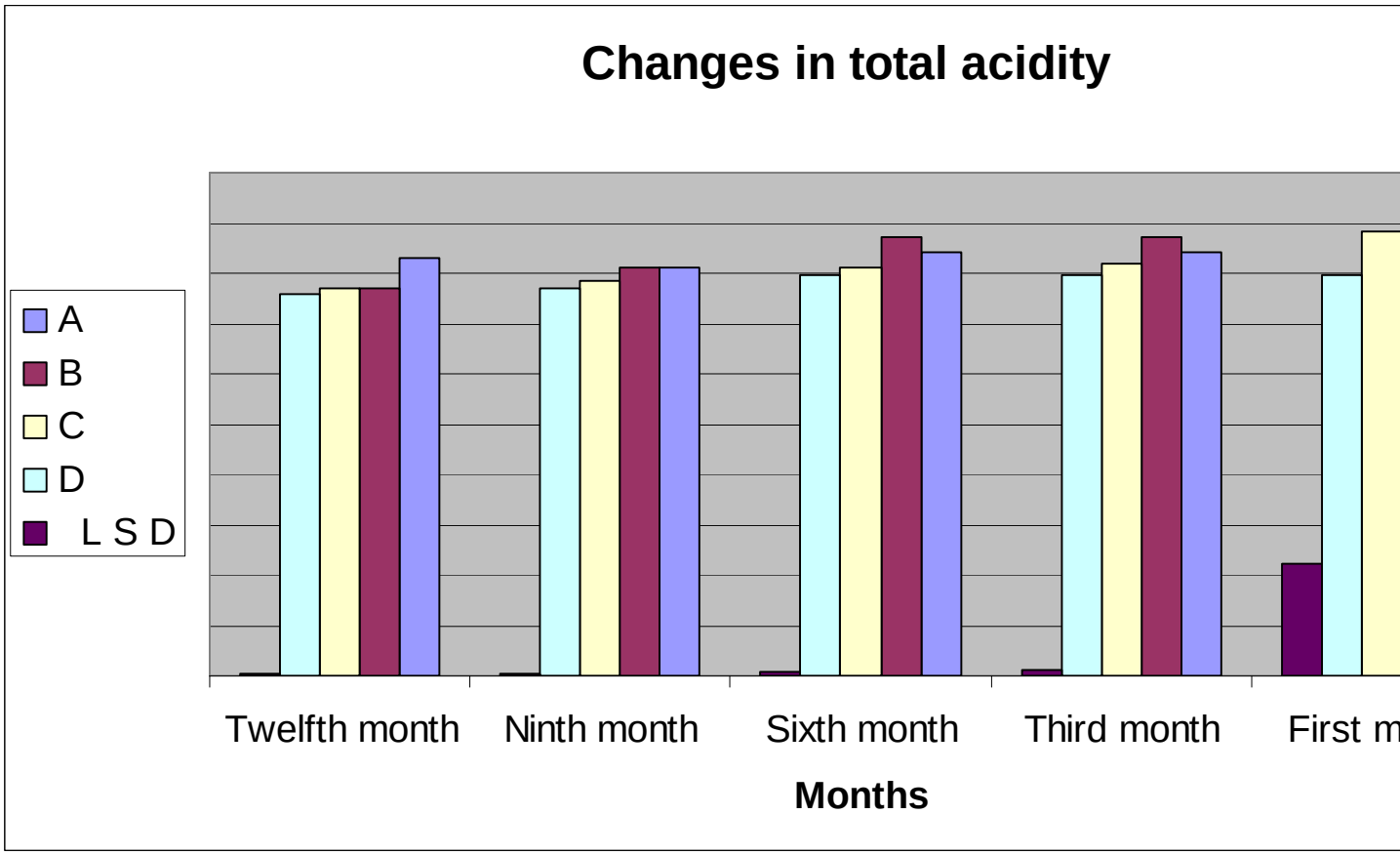
B : Liquid concentrate 39 % treated with CMC as anti cackig agent .

C : Liquid concentrate 36 % treated with gum arabic and CMC .

D : Liquid concentrate 32% without any additives .

L S D : least significant differance .

Figer (3) Changes in Total acidity of Hibiscus instant treatments stored at ambient temperature (34±4˚C).



4.4.4Changes in Colour intensity

Table 7 showed changes in colour intensity of four treatments of hibiscus instant drink (A ,B ,C ,D), packed in alumnium foil coated with plastic , at ambient temperature (34±4˚C) and stored for twelf months . Table 8 shows significant difference ($P \leq 0.05$) in colour intensity between the four treatments , A and B treatments recorded a similar highest value 1.20 C and D recorded 1.05% -1.02% respectively in the first month . A treatment showed a significant decrease ($P \leq 0.05$) in sixth and ninth months and recorded a significant increase in the end of storage period , B treatment showed a significant decrease ($P \leq 0.05$) ,during third to twelfth months of storage period , C treatment showed a significant increase ($P \leq 0.05$) in the third and sixth month and recorded significant decrease at the end of storag period .D treatment showed insignificant difference during storage periods , it recorded a lowest values of 1.02% in colour intensity whereas A treatment recorded a highest value of 1.20% .

4.4.5 Changes in Yeast content

Table 8 shows the change in yeast content for four treatments of hibiscus instant drink (A , B , C ,D) packed in aluminum foil coated with plastic , at ambient temperature (34 ±4 ˚C) A significant difference ($P \leq 0.05$)was reported in the first

Table 7 Changes in Yeast content of Hibiscus instant treatments stored at ambient temperature (34±4˚C)

Instant treatment	First month	Third month	Sixth month	Ninth month	Twelfth month
A	1.20 a	1.15 a	1.10 b	1.10 b	1.17 a
B	1.20 a	1.10 b	1.10 b	1.10 b	1.00 c
C	1.05 b	1.15 a	1.15 a	1.02 c	1.02 c
D	1.02 c	1.02 c	1.00 c	1.02 c	1.02
L S D	0.0332	0.0332	0.0465	0.066	0.148

Legend

A : liquid concentrate 40%treated with gum arabic .

B : liquid concentrate 39% treated with CMC as anti cacking agent .

C : liquid concentrate 36% treated with gum arabic and CMC.

D : liquid concentrate 32% without any additives .

L S D : least significant difference .

Table 8 Changes in Colour intensity of Hibiscus instant treatments stored at ambient temperature (34±4˚C)

Instant treatment	First month	Third month	Sixth month	Ninth month	Twelfth month
A	1x10 b	1x10 b	1x10 b	1x10 b	1x10 b
B	1x10 b	1X10 a	1x10 a	1x10 a	1x10 a
C	Nill c	Nill c	Nill c	Nill c	Nill c
D	1x10 b	1x10 b	1x10 b	1x10 b	1x10 b
L S D	0.0000466	0.0000357	0.000466	0.00023	0.000032

Legend :-

A Liquid concentrate 40% treated with gum arabic .

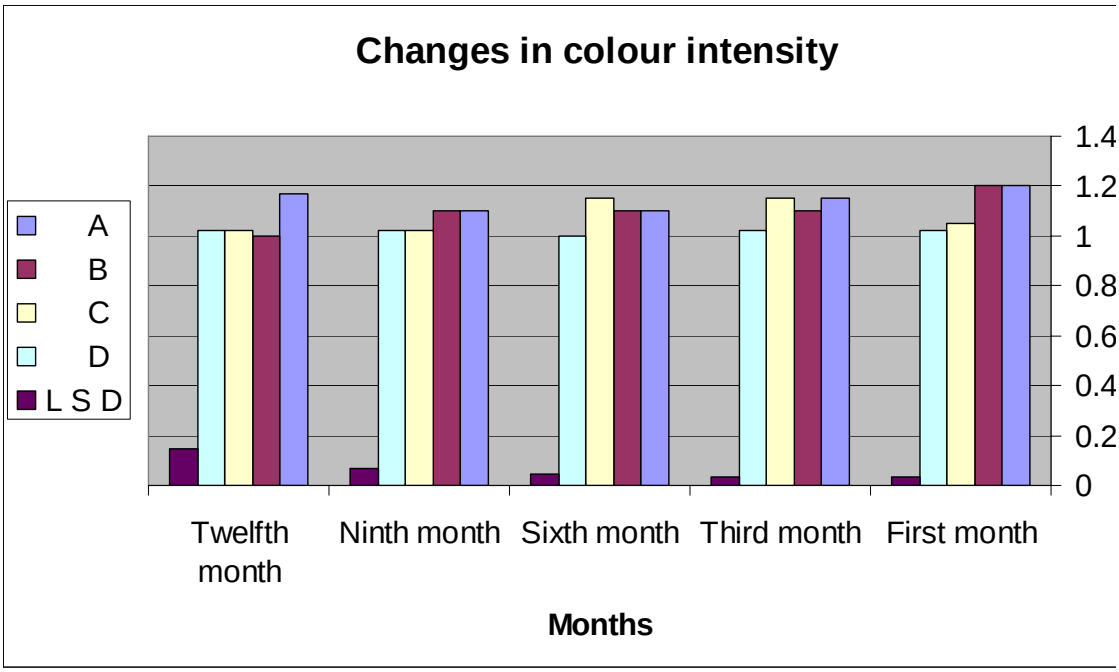
B liquid concentrate 39% treated with CMC as anti cacking agent .

C liquid concentrate 36% treated with gum arabic and CMC .

D liquid concentrate 32% without any additives .

L S D : least significant differance .

Figer (4) Changes in Colour intensity of Hibiscus instant treatments stored at ambient temperature (34±4˚C)



month . C treatment gave the lowest value compared with the other treatments A,B ,D and C showed significant difference compared with (1x 10) co/ml ,recoded by A,B and D .

Table 8 shows insignificant increase ($P \leq 0.05$) in yeast content between the beginning and end period of storage for A and D treatment , B treatment recorded significant increase in third month and insignificant changes within sixth ,ninth and twelfth month of the storage period

4.4.6 Changes in Solubility rate

Table 9 shows Changes in solubility rate per second (SR) of the four treatments of the Hibiscus instant drink (A,B.C and d) backed in Aluminum foil coated with plastic, at ambient temperature ($34 \pm 4^{\circ}C$). A significant difference ($p \leq 0.05$) in SR of the treatment in the first month of storage was observed.

The SR for A,C and D treatments were 22 ,27and29seconds these values agreed with values obtained by Ismail (**1980)** which were in the range of 18to 49 seconds.

Although A and B treatments have the same SR value at first and 12^{th} month of storage period. Table (9) shows insignificant changes ($p \leq 0.05$) for the four treatments A,B,C and D during the beginning and end of the storage period.

The high values of SR reported for C,D treatments may be attributed to the treatments with Anti caking agent agents CMC and gum arabic.

Table 9 Changes in Bulk density of Hibiscus instant treatments stored at ambient temperature (34±4˚C)

Instant treatments	A	B	C	D	L S D
First month	22.23 c	22.33 c	26.67 b	29.00 a	1.71
Twelfth month	22.41 c	21.67 c	27.00 b	29.33 a	0.76

Legend

A Liquid concentrate 40% treated with gum arabic .

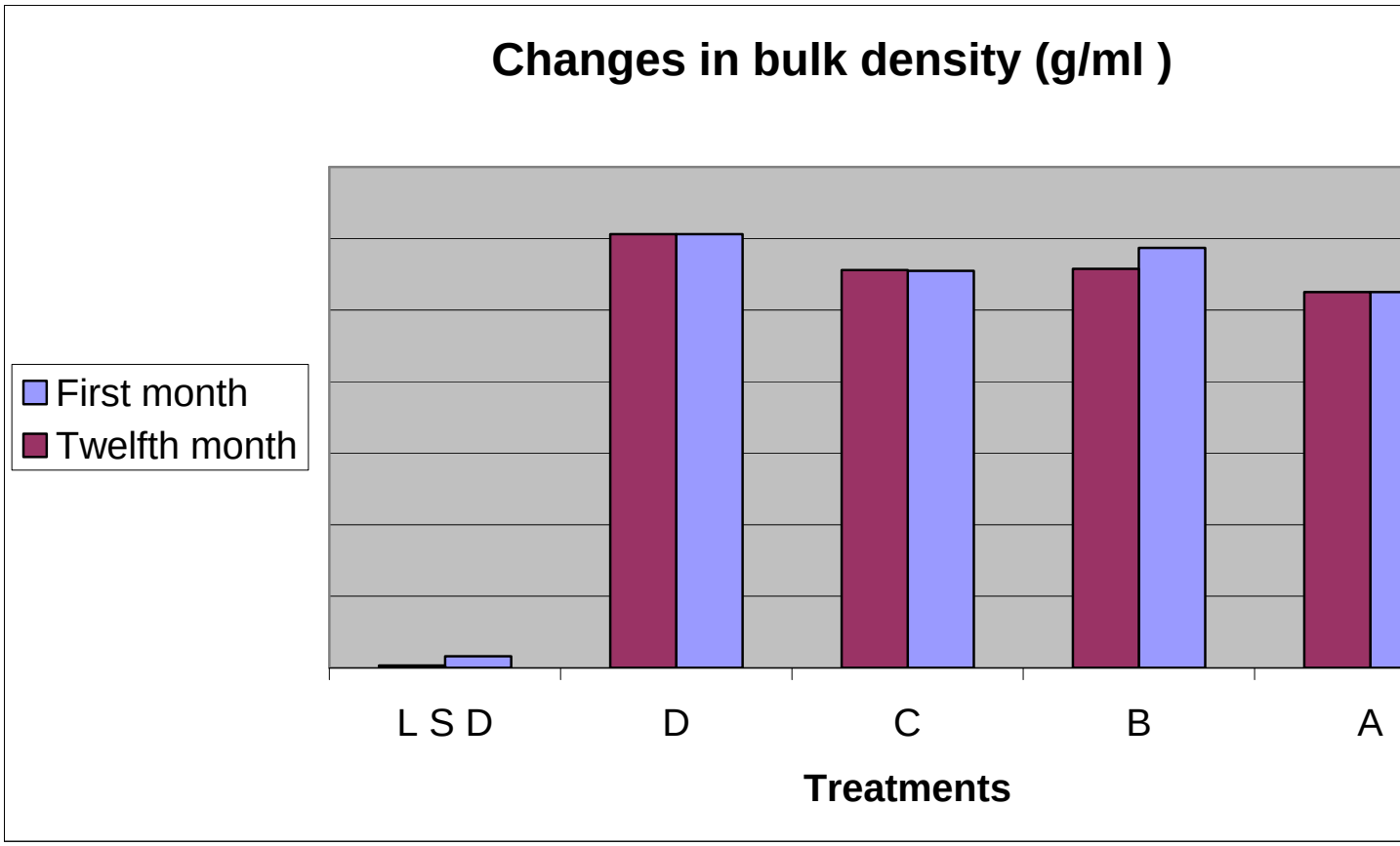
B Liquid concentrate 39% treated with CMC as anti cacking agent .

C Liquid concentrate 36% treated with gum arabic and CMC .

D Liquid concentrate 32% without any additives .

L S D : least significant differance .

Figer (5) Changes in Bulk density of Hibiscus instant treatments stored at ambient temperature (34±4˚C)



4.4.7. Changes in Bulk density

Table 10 shows changes in Bulk density of the four treatments of hibiscus instant drink (A,B,C,D) packet in Aluminum foil coated with plastic , at ambient temperature (34±4˚C). A significant difference ($p \leq 0.05$) in (B ,D) among the four treatments was showed in Table 10 , A B C and D treatment showed insignificant difference among the first month and twelfth month of storage period , D treatment recorded the lowest value of 0.606 at the beginning and end of storage this refers to the difference in concentration of hibiscus liquid and to additives used which were gum Arabic and CMC. Ismail (1980) reported similar value of 0.66 for agglomerate treated with Tri calcium phosphate (TCP) which was used as anti caking agent .

Table 10 Changes in Solubility rate of Hibiscus instant treatments stored at ambient temperature (34±4˚C)

Instant treatments	A	B	C	D	L S D
First month	0.525d	0.587 b	0.555 c	0.606 a	0.016
Twelfth month	0.525d	0.588 b	0.556 c	0.606 a	0.0036

Legend

A Liquid concentrate 40% treated with gum arabic .

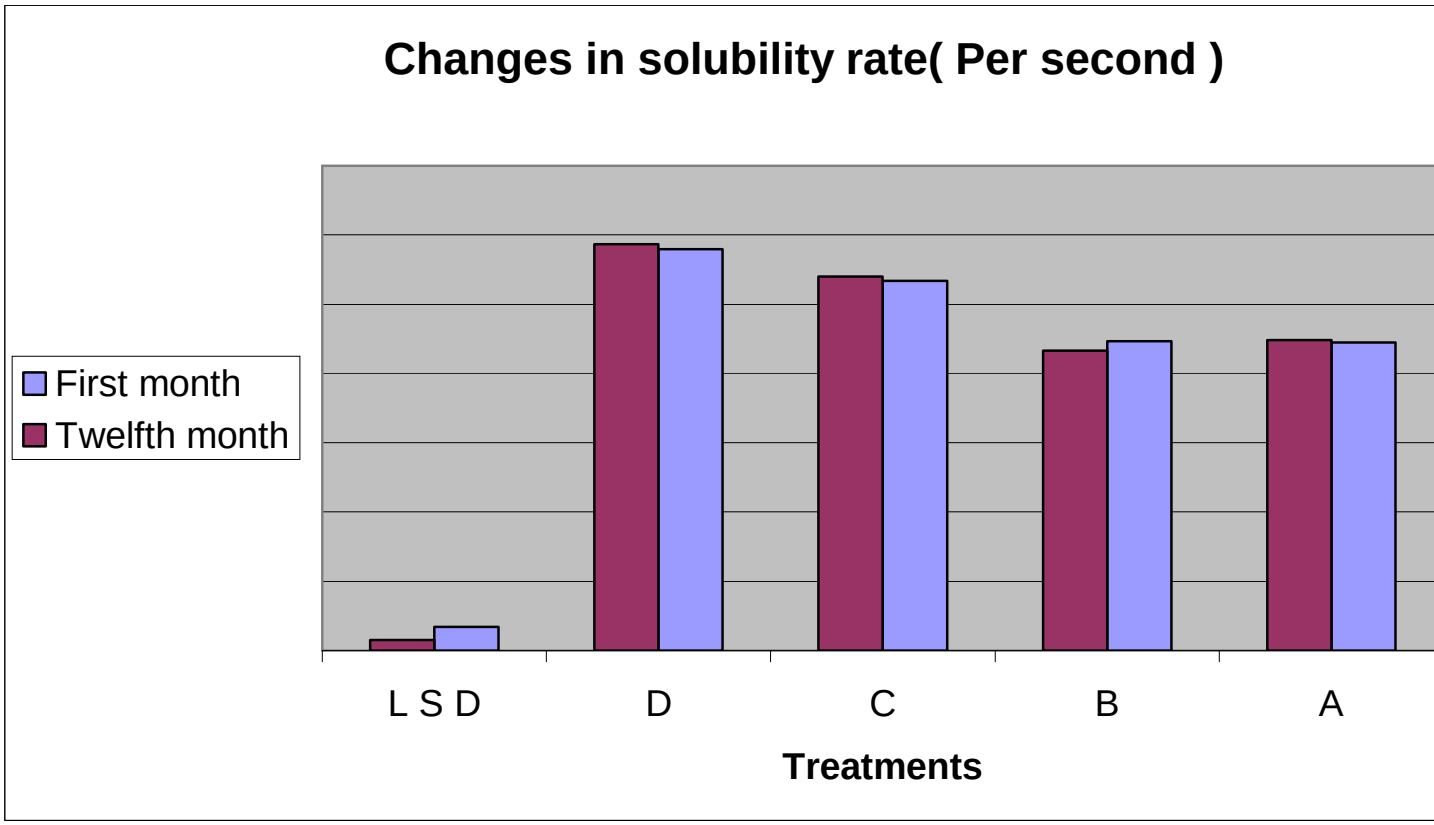
B Liquid concentrate 39% treated with CMC as anti cacking agent .

C Liquid concentrate 36% treated with gum arabic and CMC .

D Liquid concentrate 32% without any additives .

L S D : least significant differance .

Figer (6) Changes in Solubility rate of Hibiscus instant treatments stored at ambient temperature (34±4˚C)



CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

-There is a directly Porportional relationship between hibiscus concentration and particle size of instant drink granule .

-Caking is expected to exist as concentration increases.

32 brix and below is the best concentration of hibiscus extract to give the fine granulars .

-The drying process on sugar powder prevent hibiscus pigements from hydrolysis degradation due to the decrease of the hydroxyle groups (OH-)

Recommendations

*Increasing efforts towards mechanical harvesting and drying reliable relative humidity to stop yeasts growth.

*Conducting intensive studies about hydrolysis degradition of anthocyanine pigments.

*Changing the trend of raw flowers exports to concentre liquids at 40 to 45 brix in dark containers .

*Applying this process for other local products like Tamarind and Baobab to offer natural components to the existed consumtion culture

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