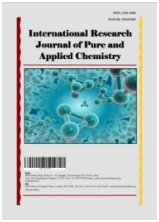

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# Extracting and Studying the Factors Affecting the Tanning Material in the Garad Pods

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***Authors' contributions***

*This work was carried out in collaboration among all authors. Author EE designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author GA managed the analyses of the study. Authors EE and GA managed the literature searches. All authors read and approved the final manuscript.*

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## ABSTRACT

In this study the chemical structure of Garad tan pods (acacia – nilotica) were determined and investigated. It was found that Garad tan is a polyphenol type of structure and consequently it has an antiseptic effect, it is also used as pretaning and retaning agent.
Garad pods need to be disintegrated and ground into fine grains of very small diameter. In its powder form it cannot be used in tannage as the grains may block the sewage, hence, it has to be leached, concentrated and spray dried.
**The factors that affect leaching of the tannins from Garad pods were studied, there are:** Temperature, solvent to solid ratio, degree of mixing and time were specified and factorial experiments were designed to study the effect and significance of these factors. The levels of these factors were (30 and 50°C), (1:5 and 1:7), (with and without) mixing and (25 and 50) minutes respectively. All these factors were found to be very significant.

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**Having determined the optimum factors their levels trials were carried out at the following levels:**
**Temperature:** 50°C
**Solid to solvent ratio:** 1:7
**Degree of mixing:** With mixing
**Time:** 50 minutes
The trials made at these conditions were found to be efficient with high yield of the extract. The extract produced was concentration which makes it ready for use in pertannage or retannage or for spray drying to powder.

*Keywords: Garad pods; tannins; leaching; polyphenol.*

## 1. INTRODUCTION

Vegetable tannins are water-soluble polyphenols that are widely distributed in almost all plant foods [1]. Phenolic compounds are potential phytochemicals, which have important health promoting properties.

Tannins are antioxidants containing nutraceuticals and are suggested to be cardioprotective, anti-inflammatory, immune-modulatory, anti-carcinogenic and anti-mutagenic [2]. It enhances glucose uptake, induce beta cell generation and increase insulin activity. Itsanti-inflammatory effect helps control gastritis, esophagistis, enteritis and irritating bowel disorders. It also heals burns and internal wounds, and is effective in protection of kidneys. Tannins have also been reported to use for immediate relief of sore throats and diarrhea [3].

Tannins are classified into hydrolysable tannins and condensed tannins. The first one is usually present in low amounts in plants (gallotannins, ellgitannins) and the condensed tannins are a group of naturally occurring polyphenolic bioflavonoids (catechins) [2].

Tea, coffee, supari, apples, dry fruits, mint etc. are rich sources of tannnis. Condensed tannins are widely distributed in fruits, vegetables and certain food grains and legume. It is reported that protective effect of tannins against so many diseases is because of its capacity to act as free radical scavengers and to activate antioxidant enzymes; it is particularly due to the condensed tannins- catechins [4].

Vegetable tanning refers to leather that is tanned with oak and spruce bark. Also quebracho, tara pods, olive leaves, rhubarb roots or mimosa are common. These substances are placed in a pit along with the skins and hides. As these tannins are derived from plants, the leather is called vegetable-tanned leather [5].

The Garad tree (*Acacia nilotica*), is one of most diverse of all the shrub species. It is a shrub that belongs to the family Fabaceae and the subfamily Mimosaceae and to the genus Acacia. The tree is called the Egyptian Mimosa in Egypt, the Garad in the Sudan and is referred to as Booni in Nigerian Yoruba language [6].

Acacia has multiple benefits to human beings. It is widely used as an ornamental tree as well as in the perfume industry. The roots of the acacia tree were boiled and used as a treatment for rabies and the feathery shoots are used in fries, soups and gravies. The honey made from the forage is flavorful and has a very smooth texture. The seeds are either eaten raw, roasted or salted. Sauce and fritters are also made from the acacia tree and more popular in Mexico [6]. An infusion make from the bark of this tree or the gum is an effective medicine for diarrhea. The bark of (*Acacia nilotica*) tree is useful in the treatment of eczema. Chewing the fresh bark of the tree daily, helps strengthening loose teeth and arrest any bleeding from the gums [7]. The leaves of the tree are effective in the treatment of conjunctivitis. The leaves of the (*Acacia nilotica*) were also found beneficial in treating epiphora that is the watering of the eyes According to Odugbemi [8]. *Acacia nilotica* can allay any irritation of the skin and smooth the inflamed membranes of the pharynx, alimentary canal and the genitor-urinary organs. The fresh pods of were found effective in treating sexual disorders such as spermatorrhea, loss of viscidity of semen, frequent night discharges and premature ejaculation. The African Zulu tribe, takes the bark of (*Acacia nilotica*), for cough treatment and also uses it for treating diarrhea, dysentery, leprosy and as an astringent. The bark or gum of the tree is used in West Africa to treat cancers and/or tumors of

ear, eye or testicles. In Senegal, the bark, leaves and young pods are chewed as an antisorbutic [7]. In Lebanon, (*Acacia nilotica*) is infused with orange flower to treat typhoid convalescence and use the roots to treat tuberculosis. The Egyptians believe that diabetics may eat carbohydrates as long as they consume powdered pods of A. nilotica. In Ethiopia, (*Acacia nilotica*) is used as a lactogogue (increase milk supply). In Australia, the bark is used for the treatment of hemorrhoids, skin eruption leg sores, mouth ulcers, sore throat and dental infections as well as discharge and excess mucus [6]. However, Abdel-Rahim and Idris [9], found that The present study was mainly designated to investigate the antifungal properties of the extracts of the different garad tree parts against both fungi; Penicillium italicum and Aspergillus niger.

The total area of the (*acacia – nilotica*) forests was estimated at about 180,000 (in 1982) acres distributed in eastern, central and western Sudan. More than 20,000 ton of pods can be collected annually from these forests, but only 10% of the available and accessible fruits are collected owing to the restricted demand and a comparatively short collecting season [10].

The Sudan is regarded as the country with the widest distribution of these plants and is the major supplier of the pods.

When Garad pods are crushed, they disintegrate into three parts, namely: The husk with about 12% pure tannins, the seeds with no tannin content and the powder with approximately 55% tannins. The seed and husk form about 63.6% of the weight of the pod, remain being the grain powder. The tree has many economic and medical uses in West Africa [10].

In the dry regions the tree is much mutilated immature pods. The seed alone crushed or made into cake are used for cattle feed.

Sudan has more than 12 mechanized tanneries with capacities ranging from 3000 to 5000 skins and from 200 to 400 hides. These tanneries used Garad grain powder for pretannage and retannage with problems of iron stains, which affect the quality of the leather, produced. The tanneries also complain that the residual grain blocks the pipes of the sewage.

In order to solve the problem garad pods had to be crushed, leached with warm water, the resulting solution had to be concentrated and spray dried. Oxalic acid added to the powder to mask the iron, which is insoluble at low pH. The leached solution has to be concentrated and evaporated. This will raise the concentrated of tannins to a level that will make its application or spray drying easier.

## 1.1 Leaching

Vegetable tannins are usually leached from crushed garad pods using water as a solvent, and then filtered, and then concentrated through evaporation in atmospheric evaporator and spray dried. The powder produced will be used in local tanneries for retannage and pretannange. This operation is one of the oldest in the chemical industries the metallurgical industries are perhaps the largest users of the leaching operation) (Taysser, 2005).

Good results are obtained by using a wooden vat and morter for pounding the leaves, pods, or fruit, small powder – driven mills (Taysser, 2005).

## 1.2 Evaporation

A solution boils in an evaporator to give off the vapor and becomes concentrated. Chemical evaporators are often built and operated as multiple – effect units for achieving steameconomy. Solution often exhibit boiling point elevation, thus reducing the temperature driving force for heat transfer [11]. Boiling point elevation is the difference of boiling point of concentrated solutions and the boiling point of pure water (Binay KD, 2001).

## 1.3 Spray Drying

Concentrated solution can be dried by spraying them as fine droplets into a stream of hot gas in spray drier. The liquid to be dried is atomized and introduced into the large drying chamber, where the droplets are dispersed into steam of heated air. The particles of liquid evaporator rapidly and dry before they can be carried to the sides of the chamber and the bulk of the dried powder which results fall to the conical bottom of the chamber to be removed by a stream of air to the dust collector.

In order to obtain rapid drying , atomization of the feed must provide small particles of high surface

to weight ratio, whose diameter is usually in the range 10 to 60 m, for this purpose, spray nozzles or rapidly rotating disls can be used. The liquid or the slurry is fed into the disk near the center and is centrifugally accelerated to the periphery from which it is thrown liquid properties and feed rates can be handled satisfactorily and even thick slurries can be atomized without clogging the device.

The drying gas, either fluegas or air, can enter at the highest practical temperature, 80°C to 760°C (175°F to 1440°F) limited only by the heat sensitivity of the product [12]. Since the contact time for product and gas is so short, relatively high temperatures are feasible. Cold air is sometimes admitted in to the drying. Chamber walls in order to prevent sticking of the product to the sides. The effluent gas may convey all the dried product out of the drier or only the fines, but in either case the gas must be passed through some type of dust collector such as cyclones or bag filters of dust. Recirculation of hot gas to the drier for purpose of heat economy is not practical, since the dust recovery operation cannot usually be accomplished without appreciable heat loss.

The dried product is frequently in the form of small hollow beads of low bulk density. Some control over the bulk density. Is usually possible through control of the particle size during atomization or through the temperature of the drying gas (increased gas temperature, causes decreased product bulk density by more extensive expansion of the drop contents). For high – density products, the dried beads can be crushed.

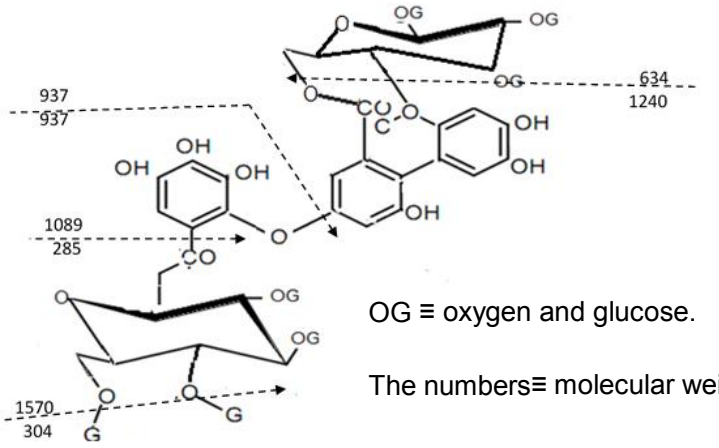
Spray drying offers the advantage of extremely rapid drying for heat sensitive product such as enzymes and vitamins (Trebal, 1984).

## 1.4 Fruits of Garad (*Acacia nilotica*)

The fruit (generally known as sunt pods garad) are an old remedy of Sudan folk medicine, but are harmless for both humans and animals, and are used mainly in tanning and dyeing. The tree is also a useful source of timber [13].

The Garad pods contain condensed tannins, together with a sufficient portion of hydrolysable tannins.

Tannin can be used in the treatment of ulcers, haemorrhoids, minor burns and frostbite as well as inflammation of the gum. Internally the powder can be administered in cases of diarrhea and alkaloidal poisoning. As a tanning material, tannins can be used in the tanning industry after certain refining by using ion-exchange resin to reduce the iron content occurring as sulphate and to comply with the international standards and specifications of commercial tanning materials. The deeply coloured compounds obtained from tannins with iron salts have been used in the manufacture of inks on a commercial scale. The use of *Acacia nilotica* in the production of native inks and dyeing of leather and fibers in different tints was based on that property. Tannins may also appear to have some




OG ≡ oxygen and glucose.

The numbers≡ molecular weight

**Fig. 1. Structure of condensed and hydrolysable tannins**

protective effects against the establishment of, and tissue damage caused by gastrointestinal nematodes [14]. Tannins levels in excess of 50 g/kg dry matter, on the other hand, can lead to low palatability, reduce digestibility, lower intake, inhibit digestive enzymes [14]. In this study the condition governing the regard the efficiency of leaching have to be investigated.

## 1.5 Factorial Experiments

Many experiment situation require the examination of the effects of varying two or more factors .it is shown that in a complete exploration of such situation it is not sufficient to vary one factor at a time, but that all combinations of the different factor levels must be examined in order to elucidate the effect of each factor and the possible ways in which each factor may be modified by the analysis of the experimental results. The effect of each factor can be determined with the same accuracy as if only one factor had been varied at a time and the interaction effects between the factors can also be evaluated.

Experiments carried out by chemists, physicists and engineers, whether in the laboratory or in the plant, are in general intended to determine the effects of one or more factors on the yield or quality of a product. The performance of a machine or a material to chemical attach, the power or fuel consumption advantage is gained if the experiment is so designed that the effect of changing any one variable can be assessed independently of the others. One way of achieving this object is to decide on asset of value, or level, for each of the factors to be studied and to carry out one or more trials of the process with each of the possible combination of the levels of the factors.

### 1.5.1 Factors

The term factor is used in a general sense to denote any feature of the experimental conditions, which may be assigned at, will from one trial to another. It may represent, for example, the temperature, pressure, or space velocity at which a chemical reaction is carried out.

### 1.5.2 Levels of factor

The various values of factor examined in an experiment are known as level.

### 1.5.3 Treatment

The set of levels of all factors employed in a given trial is called the treatment or treatment combination. This term originated in fertilizer trails, the treatment being the type and amount of the fertilizer used. The treatment combination gives a full description of the conditions under which the trials is carried out, so far us there are affected by the various factors being studied.

### 1.5.4 Response

The numerical result of a trail based on a given treatment is called the response corresponding to that treatment. The response may be the yield of a process, the purity of a chemical, the composition found for a chemical sampled and analyzed in a given way.

### 1.5.5 Effect of a factor

The effect of a factor is the change in response produced by a change in the level of the factor and measured by the difference between the average effects at the various levels.

### 1.5.6 Main effect and interaction

The average of the two methods, for example are taken over all levels of temperature and over all batches. It may be that the difference between the two methods is not the same at all temperature. The average effect is called the main effect of the factor.

If the effect of one factor is difference at difference of another the two factors, are said to interact.

### 1.5.7 The advantage of factorial design

**To sum up:**

- When there are no interactions the factorial design gives the maximum efficiency in the estimation of the effect.
- When interaction exit, their nature being unknown, a factorial design is necessary to avoid misleading conclutions.
- In the factorial closing the effect of a factor is estimated at several levels of the other factors and the conclusions hold over a wide range of conditions.

## 1.6 The Objectives

To investigate the conditions of various factors on the degree of leaching, these include:

- Solvent to solid ratio.
- Leaching temperature.
- Time of leaching.
- Degree of mixing.

To prepare an extract solutions at an appropriate composition suitable to tanning processes and spray drying.

## 2. MATERIALS AND METHODS

### 2.1 Samples Used for Experiments

Samples were taken from market, crushed and the seeds were separated.

### 2.2 Equipment Used

1. Crusher
2. Sieve
3. Refractometer
4. Glass thermometer
5. Filter
6. Sensitive balance
7. Hide powder

### 2.3 Vegetable Tannins Leaching Procedure

The crushed or chopped material is fed into a large wooden vat, coarser material being placed in the bottom where there is a perforated or lattice, loose floor. The vat is then filled with warm water and left until no more tannin will leach out. To produce stronger liquors economically a battery of several leach vats is used, as in Fig. 2 and the liquors are run on counter current basis, so that stronger liquor which has been previously extracted is run on to the largely extracted bark in order to remove the last traces of tan. A battery of six vats or more may be used all interconnected. When the bark is fully extracted or (spent) as in (C) the vat is emptied and recharged and then becomes the head leach, with (B) the bottom leach and the next to be discharged.

The water used for leaching must have a low degree of hardness and be iron-free:

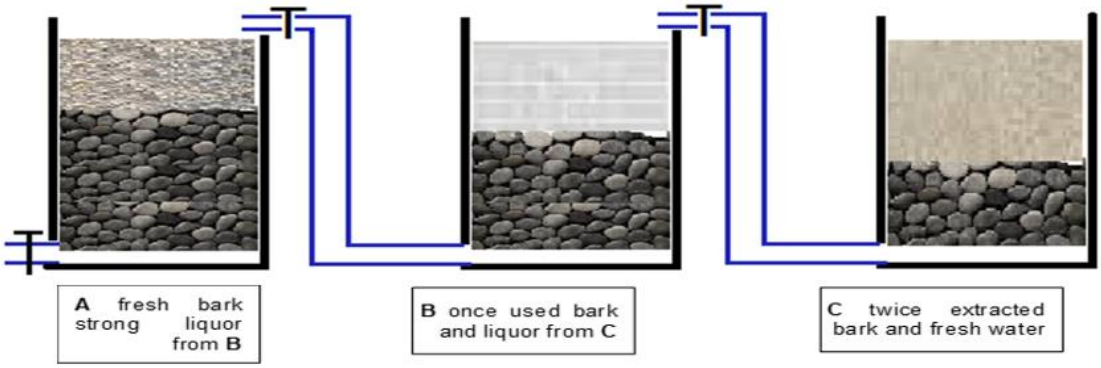



**Fig. 2. Press leach for vegetable tan**

There is an optimum temperature for each tanning material. Each of the circles stands for leaching followed by phase separation, W for leaching solvent (water), G for the solids being leached (Garad pods), E for extract solution and R for the raffinate. Subscripts refer to stage numbers.

Starting with at stage (1) a weight 100 g of crushed Garad pods is leached for half an hour with an amount of 1000 ml of water, the extract E1, is withdrawn while the raffinate R1, is extracted with afresh amount of water W in contact number (2). From stage 2, the extract E2 is used for leaching afresh amount G of crushed Garad pods in leaching (3), the raffinate R2 is leached with fresh water in stage 4 and so on. It is essential to allow enough time for drainage of all extract in order to effect a substantially complete phase separation.

The first complete cycle of the operation is represented by leaching 11 – 15. The cycles were continued until 4 consecutive extract analyses were constant within experimental errors.

## 2.4 Hydrometers

There are several types of hydrometer in use in tanneries, e.g. Barkometer Baume and Twaddell. There are all similar in appearance, consisting of a long, narrow, graduated stem attached to a weighted bulb. This is allowed to float freely in the solution, it is more buoyant the higher the specific gravity of the liquid and sinks less deeply into the liquid.

The degree to which is sinks is measured by reference to the calibrations on the stem, in term of degrees Barkometer SG. etc. it is important that the hydrometer is quite clean and floats freely. The Barkometer is most common in tanneries using vegetable tan liquors.

The relationship between S.G and Barkometer is shown in Table 1.

**Table 1. Specific gravity barkometer**

| S.G | Barkometer |
|---|---|
| 1.000 | 0 |
| 1.020 | 20 |
| 1.110 | 110 |
| 1.145 | 145 |

When using a Twaddell, the reading must be multiplied by 5 convert in to the Barkometer scale e.g.

2° Twaddell = 10° Barkometer = 1.010 specific gravity.

8° Twaddell = 40° Barkometer = 1.040 specific gravity.

Baume is widely used on the continent.

It is also used in some English tanneries where chrome liquid extracts and lime paints are prepared.

**Table 2. Calibration curve of R.I vs concentration**

| R.I | Concentration (g/L) |
|---|---|
| 0.005 | 5 |
| 0.010 | 10 |
| 0.015 | 14 |
| 0.020 | 20 |
| 0.025 | 28 |
| 0.030 | 30 |
| 0.035 | 34 |

1° Baume = 6.9 ° Barkometer.

Liquid vegetable tan extract at 200° Barkometer contain approximately 30% tans. Hence, from the Bk of the liquor in a tanning system we can calculate the approximate tan content, e.g. 100° Bk liquor will contain approximately 15% tans.

## 2.5 Analysis of Tannins

### 2.5.1 Procedure

The extract solution which mainly consists of the total solubles (tannins and non-tannins) and insoluble is first filtered. 1000 cubic centimeter is taken from filtrate for the analysis which is carried out as follows:

### 2.5.2 Total solubles

Weight out 50 ml of solution, allowed to evaporate in a water bath at 60°-70°C until completely dried, then dry in an oven at 95°-100°C to a constant weight. The total solubles is equal to this constant weight (X).

### 2.5.3 Determination of non-tannins and tannins

5 mm glass tube of 30 cm height was prepared, makeup a packing section of 20 cm which ought to be packed with chrome pre-tanned hide powder. 250 ml of the filtered solution was taken and passed to slowly over 2 hours through the hide powder, 5 ml of the raffinate solution was taken, evaporated and dried in an oven at 90° - 100°C until constant weight. This weight was the weight of the non-tannins (w), the tannins content which denoted (Z) was:

$$Z = X - W$$

## 2.6 Extract Manufacturing

It is uneconomical to produce a leach of less than 10% tan content. The leach liquors was concentrated by evaporating off the excess water to the required degree to form a liquid extract, containing 30% tan or a solid extract containing 60% tan. This required care because too high temperature might cause the tan to become darker in color, which loss of solubility and tanning content.

The vessels used for this purpose are either of copper or stainless steel. Iron and other metals suffer corrosion and discolor the tanning solution. To assist rapid evaporation and to avoid high temperature, evaporation is carried out under reduced pressure.

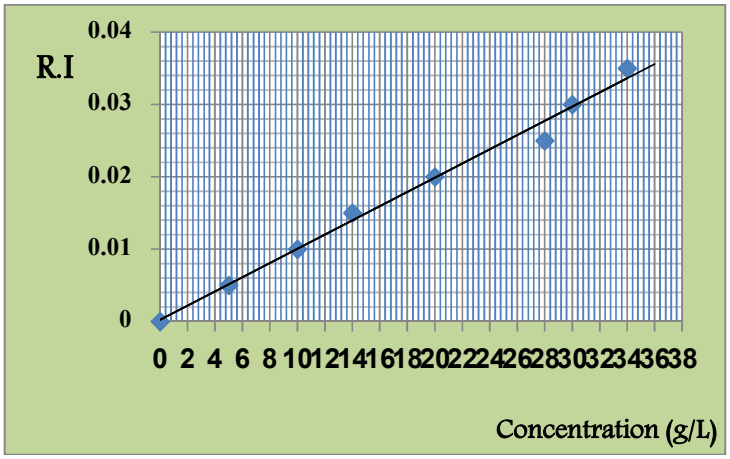



**Fig. 3. Calibration curve of R.I vs concentration**

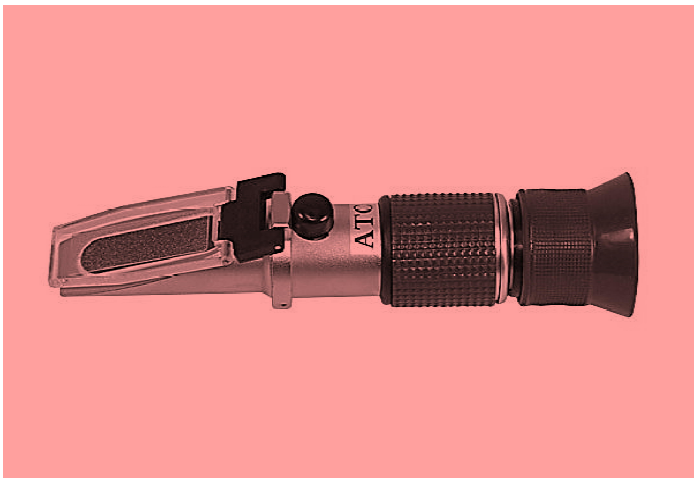



**Fig. 4. Refractometer**

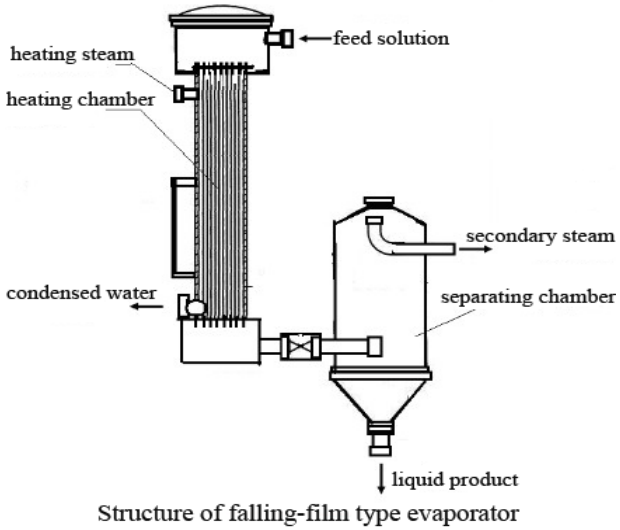



**Fig. 5. Climbing film evaporation**

## 2.7 Spray–dried Powder

To prepare (spray-dried) powders of very high tannin content (70%), liquid extracts were sprayed into the top of a very tall cylindrical chamber in such a way that they spin round the side as they fall. A very hot air draught ascends the cylinder, usually from an oil burning jet, which snap-dries the drops of liquid to powder form.

The powder was collected and bagged as it accumulates at the bottom of the cylinder (Sharp house, 1982).

## 3. RESULTS AND DISCUSSION

The procedure was applied to make the factorial experiments, 5 gm garad powder was used in each experiment.

**Table 4 shows:**

- Means all factors are at their low levels.
- a, b, c and d show high level of each factor.

ab, ac, ad, bc, bd, cd, abc, abd, bcd, acd, abcd show the interactions between factors, noting that the presence of the small letter indicates a high level of a factor while its neglecting means low level thereof.

## 3.1 Statistical Analysis

Complete Randomized design (factorial arrangement 2 x 2 x 2 x 2 ) was used in the current experiment the data was subjected to statistical analysis according to Gomez and Gomez [15] using the statistical package for social science (SPSS ) program.

The experiments were conducted under complete Randomized design (factorial arrangement). The data were analyzed by A nova according to Gomez and Gomez, [15] using the SPSS program.

With the exception of the interaction between solid to solvent ratio and temperature all these factors were found to be in significant.

**Table 3. Design of experiments**

| Treatment combination | Factors | | | | Duplicate experiments | |
|---|---|---|---|---|---|---|
| | A | B | C | D | Composition GM/L | |
| 1 | - | - | - | - | 132 | 132 |
| A | + | - | - | - | 164 | 162 |
| B | - | + | - | - | 260 | 256 |
| C | - | - | + | - | 220 | 218 |
| D | - | - | - | + | 126 | 126 |
| AB | + | + | - | - | 300 | 298 |
| AC | + | - | + | - | 240 | 240 |
| AD | + | - | - | + | 174 | 172 |
| BC | - | + | + | - | 302 | 304 |
| BD | - | + | - | + | 160 | 156 |
| CD | - | - | + | + | 142 | 140 |
| ABC | + | + | + | - | 320 | 320 |
| ABD | + | + | - | + | 176 | 172 |
| BCD | - | + | + | + | 152 | 152 |
| ACD | + | - | + | + | 144 | 146 |
| ABCD | + | + | + | + | 206 | 204 |

*A (1,a) time , 25 minute – 50 minute, B (1,b) temperature , 30C° - 50C°, C (1,c) with out - with, D (1,d) solvent to solid ratio, 1:5 and 1:7*

**Table 4. Leaching experiment**

| Factor | Treatment (1) low | Treatment (2) high |
|---|---|---|
| Temperature | Room temperature | 50°C |
| Solvent solid ratio | 1:5 | 1:7 |
| Degree of mixing | With out | With |
| Time | 25 min | 50 min |

**Table 5. Determination of mean and Std. Deviation**

| Temp | Ratio | Degree of mixing | Time | Total tannins | |
|---|---|---|---|---|---|
| | | | | Mean | Std. deviation |
| 30°C | 1:5 | With out | 25 | 132.000 | 0.000 |
| | | | 50 | 210.5000 | 54.8787 |
| | | With | 25 | 219.0000 | 1.4142 |
| | | | 50 | 240.0000 | 0.0000 |
| | 1:7 | With out | 25 | 126.0000 | 0.0000 |
| | | | 50 | 173.0000 | 1.4142 |
| | | With | 25 | 141.0000 | 1.4142 |
| | | | 50 | 145.0000 | 54.8544 |
| 50°C | 1:5 | With out | 25 | 0.0000 | 0.0000 |
| | | | 50 | 299.0000 | 1.4142 |
| | | With | 25 | 303.0000 | 1.4142 |
| | | | 50 | 320.0000 | 0.0000 |
| | 1:7 | With out | 25 | 158.0000 | 2.8284 |
| | | | 50 | 174.0000 | 2.8284 |
| | | With | 25 | 152.0000 | .0000 |
| | | | 50 | 205.0000 | 1.4142 |

**Table 6. Main effect**

| | Tannins mean | Significant level |
|---|---|---|
| **Temp (°C)** | | **.0000 **** |
| 30 | 173.312 | |
| 50 | 230. 143 | |
| **Ratio** | | **.0000 **** |
| 1 : 5 | 246.214 | |
| 1 : 7 | 159.250 | |
| **Degree of mixing** | | **.023 **** |
| With out | 181.786 | |
| With | 215.625 | |
| **Time (minute)** | | **.002 **** |
| 25 | 175.857 | |
| 50 | 220.812 | |

*** Significant at (P L 0.01)*

## 4. CONCLUSION

The interaction of solvent – solid ratio with temperature is also found to be significant as seen from Table 6. The reason for this is that the quantity of solvent with enhances the solvent capacity to each more solute.

On the other hand the temperature will make the molecules of the solute more rapidly and therefore increases the rate of mass transfer as well as the activity of the solute.

It is also observed that the mixing of the solution is significant, which eventually brings the molecules closed to each other, so the concentration of the solution is enhanced.

The time of the leaching is also significant, because leaching operation needs time to be closed to equilibrium conditions. The levels of the factors in accordance with the factorial experiments are temperature 50°C, solid - solvent ratio 1:7, degree of mixing with mixing and time is 50 minutes.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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