Extraction Rates of Oils of Some Seeds by using Different Speeds of the Centrifuge

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ABSTRACT— ***Centrifuge has been introduced as a rapid method for extraction of oil from plant parts in addition to conserve time, solvent and energy. The aim of this study is to evaluate the extraction rate of oils from some oil-seeds (Groundnut and Sesame) by using three speeds (low, medium and the high speed) of the Centrifuge (model: 80-1, Made in China) using hexane solvent, and to determine the quality characteristics of the extracted oils. The seeds were crushed and kept in plastic bags in order not to lose the oil. The estimation of the extraction percentage was depended on the weight of the sample before and after the extraction at any time for each speed. The results showed that, the extract reached mean of 45% from Groundnut seeds (100% extraction) within 18 minutes at a rate of 2.31% extract/minute, using only 18 ml of hexane. The extract reached mean of 45% from Sesame seeds (100% extraction) within 18 minutes at a rate of 1.91% extract/minute, using 18 ml of hexane. The physical and chemical properties of the extracted Groundnut and Sesame oils were relatively similar to their standards it was clear that, in all cases, the high the speed used in the Centrifuge; the high was the percentage oil extract. The study recommends continuing researches on centrifugation principle to improve oil extraction methods.***

Keywords—Extraction, Rates ,Oils Seeds , Speeds , Centrifuge

1. INTRODUCTION

Most legumes are low sources of fat with the exception of soybean and groundnut (Liu, 1997). Sesame (Sesamum indicum L.) is one of the major oil seed crops grown in the Sudan (El Naim et al., 2010). Among oil crops, sesame is one of the highest in oil content. Generally, the oil content in sesame ranges from 34 to 63% (Yermanos et al., 1972). Sesame seed is rich in fat, protein, carbohydrates, fiber and some minerals. The oil seed is renowned for its stability because it strongly resists oxidative rancidity even after long exposure to air (Global AgriSystems, 2010).

Groundnut, or peanut (Arachis hypogaea), is a species in the legume family Fabaceae, native to South America, Mexico and Central America (Gibbon and Pain, 1985). It has been known to man as an important food crop for many centuries. In the Sudan, Groundnut is an important cash crop and ranked number nine in the world (FAO, 2008). It comes first in the Arab countries, both in area and production of groundnut. Groundnut is grown mainly for its oil, protein, plant residue and seed cake (Carley and Fletcher, 1995). The oil is mainly used as cooking oil and for the production of soap, margarine and cosmetics (Anyasor et al., 2009).

A centrifuge is a piece of equipment that puts an object in rotation around a fixed axis (spins it in a circle) applying a potentially strong force perpendicular to the axis of spin (outward). The centrifuge works using the sedimentation principle, where the centripetal acceleration causes denser substances and particles to move outward in the radial direction. At the same time, objects that are less dense are displaced and move to the center (Susan and Eduardo, 2004).

There are multiple types of centrifuge. Fixed-angle centrifuges are designed to hold the sample containers at a constant angle relative to the central axis. Swinging head (or swinging bucket) centrifuges, in contrast to fixed-angle centrifuges, have a hinge where the sample containers are attached to the central rotor. This allows all of the samples to swing

outwards as the centrifuge is spun. Continuous tubular centrifuges do not have individual sample vessels and are used for high volume applications (Cole-Parmer and William, 2012).
A wide variety of laboratory-scale centrifuges are used in chemistry, biology, biochemistry and clinical medicine for isolating and separating suspensions and immiscible liquids. They vary widely in speed, capacity, temperature control, and other characteristics. Controls vary from simple electrical timers to programmable models able to control acceleration and deceleration rates, running speeds, and temperature regimes. Ultracentrifuges spin the rotors under vacuum, eliminating air resistance and enabling exact temperature control. Zonal rotors and continuous flow systems are capable of handling bulk and larger sample volumes, respectively, in a laboratory-scale instrument (Susan and Eduardo, 2004).

2. MATERIALS AND METHODS

2.1.Materials

Samples of groundnut (*A. hypogaea*) and Sesame seeds (*S. indicum*) were brought from Agricultural Research Corporation-Wad Medani, Sudan, during August 2015. Study tests were run in Faculty of Engineering and Technology, University of Gezira. The samples were prepared as two grams for each and were rolled in filter paper. Hexane solvent and Centrifuge (model: 80-1, made in China) were used for extraction of oils.

2.2.Preparation of Seeds

The healthy dry seeds were cleaned manually, and then the seeds were crushed to fine granules (less than 1 mm in size) and kept in plastic bags (to prevent absorption of oil) for extraction purposes. The samples were prepared in Faculty of Engineering and Technology, University of Gezira as two grams for each and were rolled separately in filter paper and soft tissue.

2.3.Method of Extraction

The samples were placed in the six plastic tubes of Centrifuge. A piece of cotton was put over each rolled sample within the tube, under which small perforated plastic tube was also placed. The low (1000 rpm), medium (2000 rpm) and the high speed (4000 rpm) of the centrifuge were tested. The samples were prepared in 2 g for each and were rolled carefully within filter papers and fine tissues. The total extraction period was 18 minutes for Groundnut and Sesame seeds. Two ml of the hexane solvent was dropped on the cotton pieces over each rolled seed sample (solvent volume was 1.0 ml/g seed/2 minutes). After two minutes, the centrifuge was shut off, so as to reload the hexane solvent over each sample again, and then the centrifuge was put on again. The process of on-off for the centrifuge was repeated several times till the extraction percentage was 100% at the high speed. The estimation of the oil content was depended on the weight of the sample before and after the extraction. The centrifugation process was run under room temperature ($26\pm3^{o}C$).

2.4.Physical Characteristics

2.4.1.Determination of moisture content in the seeds:

Moisture content was carried out according to AACC (1983), whereby 5 g of each sample (groundnut or sesame seeds) were weighed into a pre-dried, clean weighed porcelain dish. The samples were then placed in an air oven adjusted to $130^{o}C$ for 3 hours; the samples were then removed from the oven, and cooled in a desiccator at room temperature and weighed. Moisture and dry matter (D.M) content were calculated according to the following formula:

$$\text{Moisture content} = \frac{loss\ of\ weight \times 100}{weight\ of\ sample}$$

D.M = 100- moisture%.

2.4.2.Determination of Refractive Index:

Was measured by using Refract meter (Model ABBE60) in the laboratory of Food analysis, Faculty of Engineering and Technology, University of Gezira.

2.4.3.Determination of Density and Specific Gravity

According to (John, 2003), 10 ml of the oil (V) was measured in a pre-weighed measuring cylinder (W). The weight of the cylinder and oil were measured (W_1). The density of the water and the oil was then obtained as follow:

$$\textbf{Density of water} = W_1\text{-}W/V$$

Where:
W_1 = weight of measuring cylinder + water
W = weight of empty measuring cylinder
V = volume of water used

Density of oil sample = W_1-W/V

Where:
W_1 = weight of measuring cylinder + oil sample
W = weight of empty measuring cylinder
V= volume of oil sample used
Therefore: **Specific gravity** = Density of water / Density of oil sample

2.5.Chemical Characteristics

2.5.1.Determination of Saponification Value:

According to (Jimoh *et al.*, 2006), about 2 g of the oil sample were added to a flask with 30 ml of ethanol KOH and were then attached to a condenser for 30 minutes to ensure the sample were fully dissolved. After sample had cooled, 1 ml of phenolphthalein was added and titrated with 0.5M HCl until a pink end point has reached. Saponification value was calculated from the equation:

Saponification value = (S – B) x M x 56.1/sample weight

Where :
S = sample titer value
B = blank titer value
M= morality of the HCl
56.1 = molecular weight of KOH

2.5.2.Determination of Iodine Value (IV):

According to Jimoh *et al.* (2006), 0.4 g of sample oil was weighed into a conical flask and 20 ml of carbon tetrachloride were added to dissolve the sample. Then 25 ml of Wijs reagent were added to the flask using a safety pipette in fume chamber. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 hours and 30 minutes. At the end of this period, 20 ml of 10% aqueous potassium iodide and 125 ml of water were added using a measuring cylinder. The content was titrated with 0.1M sodium thiosulphate solution until the yellow color almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other sample. The iodine value (IV) is given by the expression:

Iodine Value = 12.69 C ($V_1 – V_2$)/M

Where:
C = Concentration of sodium
V_1 = Volume of sodium thiosulphate used for blank
V_2 = Volume of sodium thiosulphate used for determination
M = Weight of the sample

2.5.3.Determination of Free Fatty Acids (FFA):

According to AOAC (1997), 2.0 g of sample were measured into 250 ml Erlenmeyer flask, 100 ml of ethanol were added and followed by 2 ml of phenolphthalein indicator. The mixture was shaken and titrated against 0.1M NaOH with continuously shaking until the endpoint is reached, which is indicated by a slight pink color that persists for 30 seconds, the free fatty acids is expressed as:

FFA% = V x N x 282 x 100/ W

Where :
FFA% = Percent of free fatty acid (g/100g)
V = Volume of NaOH (ml)
N = Morality of NaOH
282 = Molecular weight of oleic acid
W = Weight of oil sample

2.5.4.Determination of Peroxide Value:

According to Nielsen (2003), 2.0 g of oil sample were added to 22 ml of a solution mixture of 12 ml chloroform and 10 ml acetic acid. 0.5 ml of saturated potassium iodide was added to the flask. The flask was corked and allowed to stay with occasional shaking for 1 minute. 30 ml of distilled water were then added to the mixture and titrated against 0.1M of $Na_2S_2O_3$ until yellow color is almost gone. 0.5 ml of starch indicator was quickly added and titration continued until blue color just disappeared. A blank titration was also carried out at the same condition.

Peroxide Value = (S – B) x N x1000/W

Where:
Peroxide Value = Meq peroxide per 100g of sample
S = Volume of titrate for sample

B = Volume of titrate for blank
N = Morality of $Na_2S_2O_3$ solution and W = Weight of sample

2.6.Statistical Analysis

Microsoft office, excel 2007 was used to calculate the regression model output for the obtained data. The regression analysis tool was also used to describe the relation between the observed increases in the extraction percentages (the dependent variable) in accordance to the intervals of the test period (the independent variable).

For each seed sample (Groundnut or Sesame) and for each speed (low, medium or high) of the centrifuge, R^2 (the correlation coefficient; which reflects the status of homogeneity between time and extraction percentage), intercept (the hypothetical starting point of the regression line and the expected value of Y corresponding to time zero), x-coefficient (the constant rate of increase in extraction percentage/minute) and the standard error in X variable (SE-X) and in Y variable (SE-Y) were obtained according to David (2005).

The regression line was drawn manually, using the principle described by Ray (2015); Bolker *et al*., (2008). The obtained line was used to predict time required for 100% extraction (the end point), specially for the medium and low speed treatments.

3. RESULTS AND DISCUSSION

3.1.Moisture content in groundnut and sesame seeds

The moisture content in the tested groundnut seeds ranged between 5.2% and 5.62%, while that of sesame ranged between 4.30% and 4.80%. Onwueme and Sinha, (1999), found that groundnut seeds contain 47.7% oil and 5.4% water. USDA (2011) reported that, the moisture content in sesame seeds was about 3.75%.

3.2.Oil Extraction-rate from Groundnut Seeds

Oil extraction rates from Groundnut seeds (in term of percentage) using hexane solvent in respect to time (per minutes) and different speeds (low, medium and high) of centrifuge machine, were presented in Table (4.1). At low speed, the extraction percentage was 11% after two minutes, and it reached 36% after 18 minutes. R^2 was very high (0.98), while that, the X-coefficient was 1.67 (i.e. 1.67% of the oil was extracted from groundnut seed each minute).

At medium speed, the extraction percentage was 12% after two minutes, and it reached 40% after 18 minutes. R^2 was very high (0.98), while that, 1.85% of the oil was extracted from groundnut seed each minute. At high speed, the extraction percentage was 14% after two minutes, and it reached 48% after 18 minutes. R^2 was very high (0.95), while that, 2.31% (X-coefficient) of the oil was extracted from groundnut seed each minute.

Oil content in groundnut seeds estimated to be 48% - 52% according to the variety (Christov, 2012), while it was found to be 44.8% - 51.7% in some samples of groundnut seeds obtained from Gezira State (Arabi, 2014).

It was clear that, the centrifuge machine at high speed required 18 minutes and 18 ml hexane to reach 100% extract of groundnut oil, compared to the above standards, while at the medium speed and the extraction rate of 1.85%/minute, the machine reached 83.33%, and 75% at the low speed during the same period (18 minutes) using the same volume of hexane (18 ml) (Figure (.1).

3.3.Oil Extraction-rate from Sesame Seeds

Oil extraction rates from Sesame seeds (in term of percentage) using hexane solvent in respect to time (per minutes) and different speeds (low, medium and high) of centrifuge, were presented in Table (4.2). At low speed, the extraction percentage was 5% after two minutes, and it reached 29% after 18 minutes. R^2 was relatively high (0.92), while that, the X-coefficient was 1.60 (i.e. 1.60% of the oil was extracted from Sesame seed each minute).

At medium speed, the extraction percentage was 11% after two minutes, and it reached 36% after 18 minutes. R^2 was very high (0.98), while that, 1.61% of the oil was extracted from Sesame seed each minute. At high speed, the extraction percentage was 13% after two minutes, and it reached 45% after 18 minutes. R^2 was very high (0.98), while that, 1.91% (X-coefficient) of the oil was extracted from Sesame seed each minute.

Sesame seed contains 40-50% oil (Salunkhe *et al*., 1992). It was clear that, the centrifuge at high speed required 18 minutes and 18 ml hexane to reach 100% extract from Sesame oil, compared to the above standards, while at the medium speed, the machine reached 80%, and 64.44% at the low speed during the same period (18 minutes) using the same volume of hexane (18 ml) (Figure (2).

3.4.Physical Properties of the Extracted Oils

Table (3) showed some physical properties (the density, refractive index and the specific gravity) of oil extracted from Groundnut and Sesame seeds by using centrifuge and hexane solvent. The density of Groundnut oil was 0.90, the refractive index was 1.4650 and the specific gravity was 0.92 which was relatively similar to the CoDEX-STAN210 (1999) standards of (0.912–0.920), (1.460-1.465) and (0.910-0.915), respectively, therefore, the extracted Groundnut oil has good quality.

The densities of Sesame oil was 0.88, the refractive index was 1.468 and the specific gravity was 0.91, while the CoDEX-STAN210 (1999) standards ranged between 0.915–0.924, 1.469-1.479 and 0.916-0.921, respectively, therefore, sesame oil extracted by hexane solvent has good quality.

The extraction of Groundnut and Sesame oils by centrifuge using hexane solvent led to yield oils with competent quality.

3.5.Chemical Properties

Table (4) showed some chemical properties (saponification value, iodine value, peroxide value and free fatty acid) of Groundnut and Sesame oils extracted by using centrifuge and hexane solvent. The results showed that, in Groundnut oil, the saponification value was 168, the iodine value was 85, free fatty acids (FFA) was 0.64, and the peroxide value was 1.5, while, the standards of Groundnut oil of CoDEX-STAN210 (1999) were: saponification (187-196), iodine value (86-107), Free Fatty Acid (0.6 max), and peroxide (1-10%).

The results of Sesame oil, the saponification value was 186, the iodine value was 102.3, free fatty acids (FFA) was 0.56, and the peroxide value was 1.5, while, the standards of Sesame oil of CoDEX-STAN210 (1999) were: saponification (186-195), iodine value (104-120), Free Fatty Acid (0.6 max), and peroxide (1-10%).

Although the moisture content in groundnut and sesame seeds, the physical and the chemical parameters were not identical with the proposed standards, but the differences (either significant or not significant) may be referred to differences in varieties and storage and preparation factors.

4. CONCLUSIONS AND RECOMMENDATIONS

4.1. Conclusions

- The centrifuge at high speed required 18 minutes and 18 ml hexane to reach 100% extract of groundnut oil, compared to the standards, while at the medium speed and the extraction rate of 1.85%/minute, the machine reached 83.33%, and 75% at the low speed during the same period (18 minutes) using the same volume of hexane (18 ml).
- The centrifuge at high speed required 18 minutes and 18 ml hexane to reach 100% extract from Sesame oil, compared to the standards, while at the medium speed, the machine reached 80%, and 64.44% at the low speed during the same period (18 minutes) using the same volume of hexane (18 ml).
- The extraction rate was speed dependent (i.e. high extraction rate was at high speed and vice versa).
- The physical properties of Groundnut and Sesame oils extracted by centrifuge and hexane solvent were relatively similar to their standards.
- The chemical properties of Groundnut and Sesame oils extracted by centrifuge and hexane solvent were relatively similar to their standards.

4.2. Recommendations

1- The study recommends continuing researches on centrifugation principle to improve oil extraction methods.

2- The extraction rate affected greatly with the sample size, which required further study.

Table.1. Percentage oil extracted from Groundnut seeds using hexane solvent in respect to time and different speeds of centrifuge

Time (minutes)	Low speed	Medium speed	High speed
2	11	12	14
4	13	14	18
6	15	20	19
8	18	21	22
10	25	24	24
12	28	32	36
14	30	35	42
16	33	37	45
18	36	40	48
Mean	**23.2**	**26.1**	**29.8**
Regression Analysis			
R-square	0.981	0.979	0.947
X-coefficient	1.67	1.85	2.31
Intercept	6.56	7.61	6.69
SE-X	0.087	0.102	0.205
SE-Y	0.986	1.148	2.309

Table.2: Percentage oil extracted from Sesame seeds using hexane solvent in respect to time and different speeds of centrifuge

Time (minutes)	Low speed	Medium speed	High speed
2	5	11	13
4	8	13	15
6	12	15	18
8	18	21	22
10	24	26	26
12	26	27	30
14	26	30	32
16	28	32	38
18	29	36	45
Mean	**19.6**	**23.4**	**26.4**
Regression Analysis			
R-square	0.919	0.979	0.984
X-coefficient	1.60	1.61	1.91
Intercept	3.56	7.36	7.36
SE-X	0.179	0.089	0.092
SE-Y	2.014	1.005	1.035

Table .3: Some physical properties of Groundnut and Sesame oils extracted by centrifuge using hexane solvent

Property	Groundnut		Sesame	
	Sample	**CoDEX-STAN210, (1999)**	**Sample**	**CoDEX-STAN210, (1999)**
Density	0.90	0.912-0.920	0.88	0.915-0.924
Refractive index	1.4650	1.460-1.4650	1.4680	1.469-1.479
Specific gravity	0.92	0.910-0.915	0.91	0.916-0.921

Table.4: Some chemical properties of Groundnut and Sesame oils extracted by centrifuge using hexane solvent

Property	Groundnut		Sesame	
	Sample	**CoDEX-STAN210, (1999)**	**Sample**	**CoDEX-STAN210, (1999)**
Saponific. value	168	187-196	186	186-195
Iodine value	85	86-107	102.3	104-120
Free fatty acid	0.64	0.6 max	0.56	0.6 max
Peroxide value	1.5	1-10%	1.5	1-10%

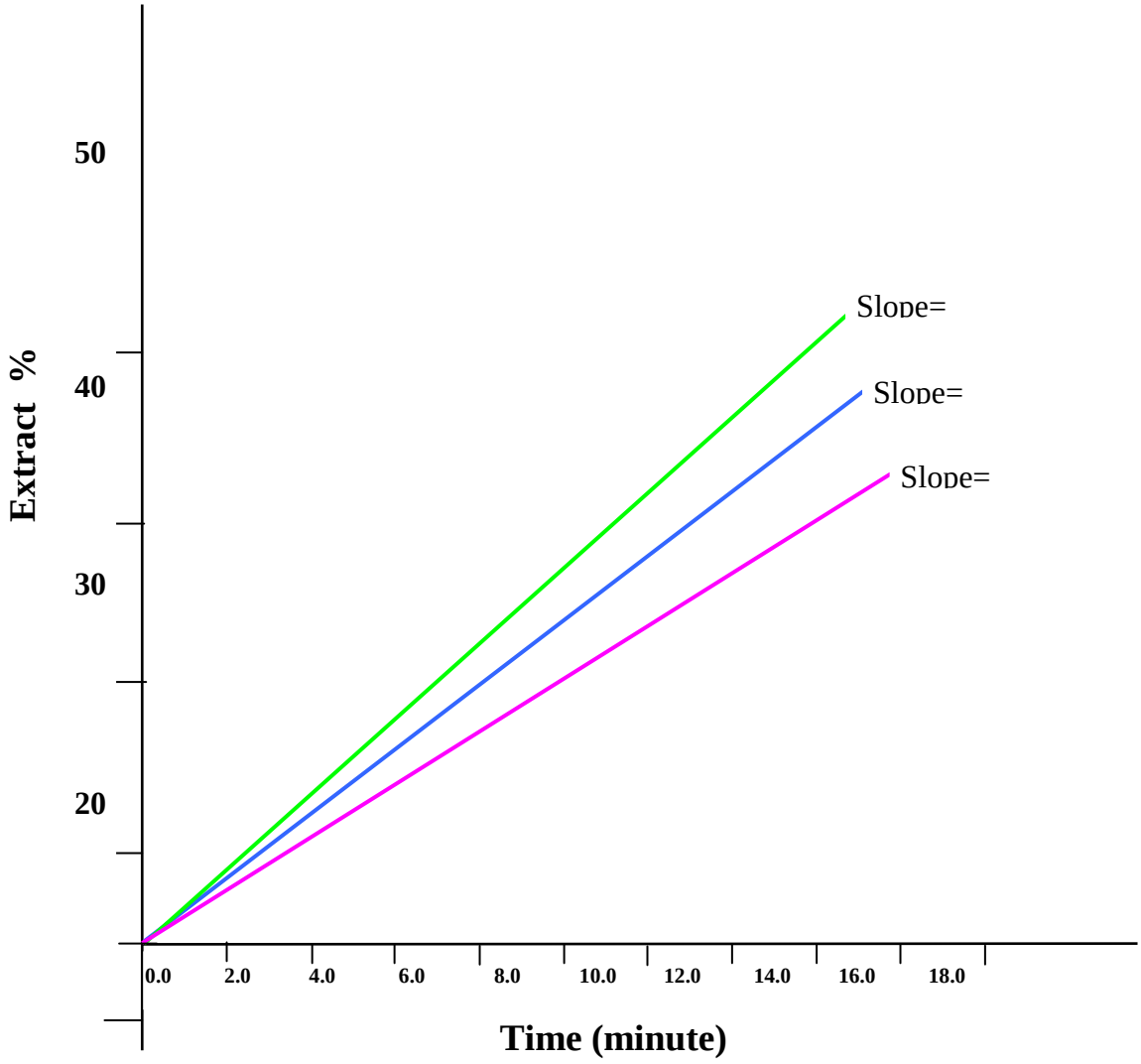

Figure.1. Regression lines of oil extracted percentage from Groundnut seeds using hexane solvent in respect to time and different speeds of centrifuge

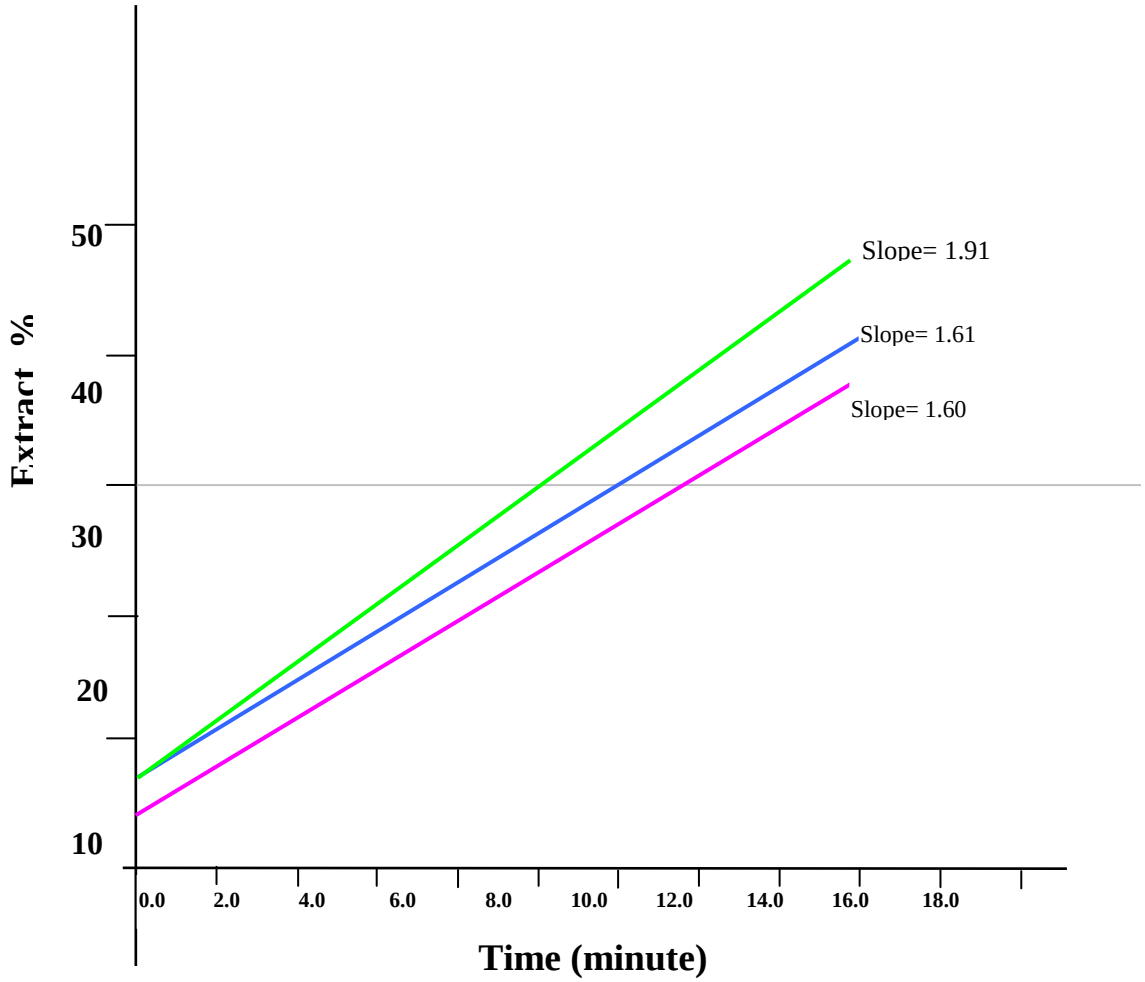

Figure.2: Regression lines of oil extracted percentage from Sesame seeds using hexane solvent in respect to time and different speeds of centrifuge

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