

2-Experimental and method

2-1-Chemical:

2-1-1- Material:

- n-hexane
- Ethanol
- Hydrochloric acid
- Potassium thiosulphate
- Potassium anhydride
- Potassium iodide
- Chloroform
- Acetic acid
- Sodium carbonate
- ether
- Wij's solution

2.1.2-Apparatus:

Conical flask ,Measuring cylinder(20,50,100), Burette (50),Glass rode ,Beakers(100,50,250),Glass watch, Funnel.

2.1.3-Instrumental:

- soxhelt
- Hot plate
- Distillation

2.2-Method:

2.2.1-Castor beans processing:

-Clearing: the castor beans had some foreign materials and dirt which was separated by hand picking.

- drying : the cleaned beans were sun dried in the open , until the casing splits and sheds the seeds, the beans were further dried in the oven at 60°C for 7 hrs to a constant weight in order to reduce its moisture at about 5-7%.

- winnowing : the separation of the shell from the nibs(cotyledon) was carried out using a tray to blow away the cover in order to achieve very high yield.

- Grinding (size reduction): mortar and pestle were used to crush the beans into a paste (cake) in order to weaken or rupture the cell walls to release castor fat for extraction.

2.2.2-Soxhlet Extraction:

300ml of normal hexane was poured into a round bottom flask. 10g of the sample was placed in the thimble and was inserted in the centre of the extractor. The Soxhlet was heated at 60°C. When the solvent was boiling, the vapour rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the centre, which contains the solid sample to be extracted. The extract seeps through the pores of the thimble and fills the siphon tube, where it flows back down into the round bottom flask. This was allowed to continue for 30 minutes. It was then removed from the tube, dried in the oven, cooled in the desiccators and weighed again to determine the amount of oil extracted. Further extraction and previous weight becomes equal. The experiment was repeated by placing 5g of the sample into the thimble again. The weight of oil extracted was determined for each 30 minutes interval. At the end of the extraction the resulting (miscella) containing the oil was heated to recover solvent from the oil. And then distilled this mixture.

2.2.3-viscosity:

the viscometer was cleaned and dried, placed in to a holder and inserted to a constant temperature bath set at 29c and then filled by sample and allowed approximately 108000 second for the sample to the bath temperature at 29c. the suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The efflux time by timing the flow of the sample as it flow freely from the upper timing mark to the lower timing mark was recorded

2.2.4- Specific Gravity:

The bottle of 25ml capacity cleaned ,dried and weighed(W_0) and then filled with the oil, stopper inserted and reweighed to give(w_1). The oil was substituted with water after washing and drying and bottle weighed to give (w_2). The expression for specific gravity (SP.gr) is : $SP = (W_1 - W_0) / (W_2 - W_0) = \text{Mass of the substance} / \text{Mass of an equal volume of water}$.

2.2.5-Moisture:

40g of the cleaned sample was weighed and dried in an oven at 80C for 7hrs and the weight was taken every 2hrs. the procedure was repeated until a constant weight was obtained. After each 2hours, the sample was removed from the oven and placed in the desiccator for 30 minutes to cool. It was then removed and re-weighed. The percentage moisture in the seed was calculated from the formula: $\text{Moisture} = 100(W_1 - W_2) / W_2\%$, where W_1 = Original weight of the sample before drying; W_2 = Weight of the sample after drying

2.2.6-Refractive index:

Few drops of the sample were transferred in to the glass slide of the refractometer. Water at 30 C was circulated round the glass slide to keep its temperature uniform. Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the scale pointed to the refractive index. This was repeated and the means value noted and recorded as the refractive index.

2.2.7-PH Value:

2g of the sample was poured in to a clean dry 25ml beaker and 13ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was then cooled in a cold-water bath to 25C. the PH electrode was standardization with buffer solution and the electrode immersed in to the sample and the PH value was recorded.

2.2.8-Percentage of Castor Oil Extracted:

20g of the sample was placed in the thimble and about 150ml of normal hexane was poured in to the round bottom flask. The apparatus was heated at 60C and allowed for 3hrs continuous extraction using Soxhlet apparatus. The experiment was repeated for different weights of the sample ,10g,15g,25g At the end,the solvent was distilled and the percentage of oil extraction was determined.

2.2.9-Acid Value:

About 0.5g of oil was weighed in a conical flask,1ml of ethanol and ether were added by measuring cylinder shake well and titrated against standard potassium hydroxide solution. (V1) the titrated repeated without the sample (blank titration) (V2). Acid value was calculated as: $(V2-V1)5.61/W$.

2.2.10-Iodine Value:

About 0.2g of oil was weighed in a conical flask, 10ml of chloroform, 25ml of wij's solution were added, the conical flask was covered and leaved it on the dark for 30minutes(the solution steered times during this period). 10ml of potassium iodide solution 10% and 100ml of distilled water were added ,the liberated titrated against 0.1N sodium thiosulphate solution using starch as indicator.(V1) the titration was repeated without the sample (blank titration)(V2). Iodine value was calculated as: $(V2-V1) \cdot M.12.69/W$ g/100 g .

2.2.11-Saponification Value:

About 0.5g of oil was weighed in a conical flask, 2.5ml of alcoholic potassium hydroxide (0.5M), reflex in water bath for about 45minute cooled and titrated against hydrochloric acid solution (0.5M) until the end point using Ph.Ph

indicator.(V1) The titration was repeated without the sample(blank titration)(V2).saponification value was calculated as: $(V2-V1) \cdot (28.05)/W$.