Comparative Studies On Graham Glass Condensing Model Types “As” And “Bs” For Extraction Of Natural Dyes From Lawsonia Inermis

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Abstract: This study was undertaken with a view to investigating the effect of the number of bulbs on the performance Graham outer jacket glass condensers. AutoCAD was used to design Graham condensers varying the number of bulbs from one to ten (1-10) on the outer jacket, while maintaining the following specifications: outer bulbs size of 048 mm, distance from bulb to bulb 18mm,inner tube length 0 9/679 mm, outer tube length 0 40/720, outlets and inlets 0 9/1.5 mm and the length of quick fit 84 mm. Ten (10) assorted pieces of each type “As” and “Bs” of this modified structures of Graham condensing models were fabricated. For types “As” Graham condensers, the outlet and inlet tubes were joined to their jackets while for the type “Bs” Graham models, the outlet and the inlet tubes were joined directly to their inner tubes. The performance of these condensers were evaluated using extraction protocol involving leave of lawsonia inermis (natural dye) acetone as solvent. Results of extraction obtained showed types “As”Graham condensing models gave the highest yield of crude extracts of 43.00% and types “Bs” condensing models with 7 bulbs yielded 41.35%.

Keywords: Graham Condenser, Extraction and Lawsonia Inermis

1.1 Introduction

Condensers are devices designed to separate one or more components of a vapour mixture by reducing the gaseous vapour to its liquid form; this condensers uses the principle of heat transfer. A heat exchanger is a device that is used to transfer thermal energy (enthalpy) between two or more fluids, (from one medium to another), or between solid surface and a fluid, or between solid particulates and a fluid, at different temperatures and thermal contact. In heat exchangers, there are usually no external heat and work interactions [1][2]. The science of heat transfer predicts that energy transfer which takes place between material bodies as a result of temperature gradient is known as thermodynamics. One of the most important fields of thermodynamics is heat transfer which relates to transfer of heat between two media. The concept of heat transfer is used in a wide range of devices like heat exchangers, evaporators, condensers, radiators, coolers and heaters. Thermodynamics also involves the study of various types of power plants like thermal plants, nuclear power plants, hydroelectric power plants based on renewable energy sources like solar, wind, geothermal and water waves[1][3][4]. Graham condenser (also Grahams or Inland Revenue condenser) has a coolant-jacketed spiral coil running the length of the condenser serving as the vapour/condensate path. This is not to be confused with the “coil condenser”. The coiled condenser tubes inside will provide more surface area for cooling and for this reason it is most favourable to use but the drawback of this condenser is that as the vapours get condensed, it tends to move them up in the tube to evaporate which will also lead to the flooding of solution mixture. The Graham Condenser consists of an inner coil surrounded by an outer tube. The coolant flows through the outer tube and the substance condenses inside the inner coil. Graham Condensers should not be confused with Coil Condensers [5]. It has been suggested that Graham Condensers are useless they cannot be used for normal distillations because the condensed liquid doesn’t flow to the receiving flask, ending up “clogging” the path. The pressure starts to rise in the apparatus, trying to push the liquid forward; this might make joints pop off or other sort of damage, they can’t be used in refluxes or either position due to the narrow path of the inner coil, pressure rises too much and vapour starts shooting out from the top of the condenser. The only time they can actually be useful is in distillations where the condenser is held vertically [6][7]. The Graham Glass condenser consist of outer glass tube usually has two hose connections, and a coolants (usually tap water or chilled water/antifreeze mixture) is passed through it. For maximum efficiency, and to maintain a smooth and correctly directed thermal gradient so as to minimise the risk of thermal shock to adjacent glassware, the coolant usually enters through the lower fitting and exists through the higher fitting. Maintaining a correct thermal gradient (entering coolant at the cooler point) is the critical factor. Multiple condensers may be connected in series. Normally a high flow rate is not necessary to maintain a cooling surface [8][4].
1.2 Extraction
Frequently, organic chemists must separate an organic compound from a mixture of compounds, often derived from natural sources or products of synthetic reactions. One technique used to separate the mixture compounds is called extraction. Extraction is a process that selectively dissolves one or more of the mixture components into an appropriate solvent. The solution of these dissolved compounds is often referred to as the extract. Extraction process include removal of soluble compounds from a solid matrix, such as in the brewing of coffee or tea, or in decaffeinating coffee with liquid carbon dioxide. In the organic chemistry laboratory, however, extraction almost always refers to the transfer of compounds from one liquid solvent to another [9].

3 Solvent Extraction
Nature rarely provides a raw materials in a pure form and in a few processes produce pure products. Because of this thoughtless provision of nature and the ineptitude of chemical synthesis, physical separation has become the most important single industrial process, the petroleum, chemical, and petrochemical industries, in general, have large investments in separation equipment and spent even more in operation. The more complicated our industrial economics becomes, the more different are the material required in a relative pure form for feedstock, and the more finished from by-products and impurities [10]. In definition, separations considered here are those separations where mixtures are divided into two pure compounds or at least into two or more fractions having different compositions by processes sometimes classified as “diffusional or mass transfer” operation. Common amount these processes are fractional distillation, fractional crystallization, adsorption, evaporation, absorption and solvent extraction [11]. A compound can be separated from impurities in a solution by extracting the compound from the original solvent into a second solvent. For the process to be selective, the compound must be more soluble in the second solvent than in the first solvent. Additionally, the two selected solvents must be immiscible, or not soluble in one another, so that they produce two separated solvent layers. After dissolving the mixture in the first solvent, the solution is added to a second solvent. The two layers are vigorously mixed to maximize the surface area between them. This mixing facilitates the transfer of a dissolved compound from one solvent layer to another. Once the transfer process is complete, the layers are allowed to form again [12][10].

1.4 Natural dyes
Natural dyes are dyes or colorants derived from plants invertebrates or minerals. The majority of natural dye are vegetable dye from plant sources, roots, berries bark leaves and woods; and other organic sources such as fungi and lichens. Natural dyes are friendly and convenient to use. They are familiar sources that can spark creative ideas and widen ones view of the world in experimenting with them. Colours can be coaxed from all kinds of natural sources. It is important to note that all natural dye recipes are safe and non-toxic [13][14]

2.0 METHODOLOGY

2.1 Design of Condensers
According to [15], Proper design or selection of condensers are very important factors for satisfactory performance of cooling systems which is based on the industrial design principles. Conventionally, the design and lengths specification for glass condensers in practice range from 120mm to 600mm [16]. AutoCAD was used to design Graham condensers by varying the number of bulbs from one to ten (1-10) on the outer jacket; while keeping the lengths (720mm) constant. Ten (10) pieces of types “As” and “Bs” of this modified structures of Graham condensers were fabricated with quick-fit and their accessories (type “As” conventional and type “Bs” unconventional). For types “As” Graham condensers, the outlets and inlet tubes were joined to their jackets while for the types “Bs” Graham models, the outlet and the inlet tubes were joined directly to their inner tubes. The difference in design and operation of types “As” Graham condensers and types “Bs” Graham condensers is the coolant flow in the outer tube for the types Graham condensers while for type “Bs”, the coolant flows in the inner tubes [4]. Fabrication was done at Scientific Equipment Development Institute (SEDI), Minna, Niger State Nigeria. Standard method of scientific glass technology fabrication of condensers was adopted used suitable tools [17].

2.2 Fabrication of Graham Condensing Models
About 48” length of 6mm diameter glass tubing and mandrel covered with ceramic paper are essential for the coil winding. The mandrel was preheated slightly and the hook was bent on the end of the 6mm tube. The hook was knot to the rod attachment on the mandrel. The 6mm glass tubing was heated in a large bushy flame until it reaches working temperature to be bend and coiled while the spacing was carefully observed. The hook end of the coil was broken away from the mandrel as soon as the coil and mandrel were cooled. The glass coil was removed from the mandrel while synchronizing the rotation and running them under cool tap water. The ends of the coil were cut, fire polished and flame annealed. The same principles of blowing bulbs was employed in blowing of the bulbs on the outer jacket for the twenty assorted pieces of types “As” and “Bs” of condensing models. The performance of these condensers was evaluated using industrial acetone grade (CH₂COCH₃) for extraction of natural dye from Lawsonia inermis (lallebature) plant leaves with voucher number 3004 which was identified in the Herbarium unit of Department of Biological Sciences, ABU, Zaria, Kaduna State.
2.3 Extraction of natural dye

The modified Graham condensing models were used to carry out solvent extraction of *Lawsonia inermis* (lallebature) plant leaves. The leave was washed with water to removed dirt and other adhering materials, then dried in a tray drier at 40°C for 48 hours and finely ground with the help of a grinding machine. 20g of the ground *Lawsonia inermis* (lallebature) was wrapped in thimble, placed inside 500ml soxhlet extractors. The four soxhlet extractors were fitted or connected with four 500ml round bottom flasks which served as the reservoir for the concentration. About 300ml of the solvent (acetone) was poured through the soxhlet opening to allow the samples and the solvent to saturate in the extracting chamber. The four sets of the condensers were connected vertically under reflux position at 50°C and operated concurrently, the temperature was allowed to gradually build up to boiling point of the organic solvent and extraction time of 6 hours was set for all the samples. Acetone was used as the organic solvent and water as the coolant flowed through the hose which was connected from the water source to the inlet tube; and another hose was connected in series to the outlet tube for the four extraction set-up. Heat was supplied from four electric heating mantles and regulated at a constant temperature of 56°C to that of the boiling point of the organic solvent 56°C. The extract continued to siphon into the reservoirs for six hours till the solvent in the soxhlet extractor chambers was colourless from the initial green colour. Extraction is said to be completed when the solvent in the Soxhlet extractor is colourless [19][12]. The flasks were rinsed properly with fresh acetone then transferred from the reservoirs into collecting sample bottles placed on top of water bath set at 50°C below the boiling point of the solvent. This was allowed to evaporate the remaining solvent leaving only the crude extract which was weighed using analytical balance.

2.4 Results of Extraction

Bar charts Figures 1-3 showed the performances of individual condensing models of both types “As” and “Bs” of Grahams. Whereby $T_A$G type “As” Graham, $T_B$G type “Bs” Graham.

![Plate I: Samples of type “As” Graham Condenser (conventional)](image1)

![Plate II: Samples of type “Bs” Graham Condenser (unconventional)](image2)

Characerization of the Condensers

Soxhlet extraction method EPA 2701C [18] was adopted for the extraction of natural dye from *Lawsonia inermis* (Lallebature) plant leaves at the research Laboratory of Glass and Silicate Technology Department of Ahmadu Bello University, Samaru, Zaria.
2.5 RESULT AND DISCUSSION

Percentage yield of extraction

Graham condensing models “As” and “Bs” show percentage yields of the crude extracts at different number of bulbs from 1-10. Graham condensing models, type “As” presented in Figure 1 with 7 bulbs gave 43.00% yields of extract followed by condensing model with 4 bulbs 37.90% while the lowest percentage of extract with 32.05% was observed in condensing model with 1 bulb. This abnormal behaviour observed in condensing model with one bulb might have resulted from the prolonged contact with the coolant which is in agreement with [6]. In Figure 2 of Graham types “Bs” condenser with 7 bulbs gave the highest percentage of extracts 41.35% followed by condensing model with 10 bulbs yield of 39.60% and the least yield of crude extracts was obtained with condensing model with one bulb 33.95% which is in conformity with the results obtained in types “As” Graham condensing model with one bulb. With the modification of Graham condensing models by introduction of bulbs at the outer jacket which increases the surface area of the condensers, it was generally observed that it disagree with the finding of [6][7], that it cannot be used in refluxes due to the narrow path of the inner coil. Figure 3 shows the overall performance of the models which established their effectiveness and optimum extraction, which was found on the condensing models with numbers of 7 bulbs with the highest yields of crude extracts. Further fabricating of these condensing models with bulbs above or below 7 will amount to waste of production, cost, materials and time.

2.6 Conclusion

The following conclusions are drawn from many other factors that might have affected the results in one way or the other such as inaccurate constructions of the condensers since these were manually fabricated. Twenty assorted types “As” (conventional) and “Bs” (unconventional) Graham condensing models were designed and fabricated. The efficacy of these condensers was established using extraction of natural dyes from lawsonia inermis (lallebature) plant leaves. The highest yield of crude extracts was obtained in Graham condensing models with 7 bulbs in both types “As” and “Bs” with 43.00% and 41.35% yields respectively.

REFERENCE


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