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Research Article

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Optimization of Graham Glass Condensing Models for Extraction of Natural dye and Rate of Solvent Recovery Process

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Abstract This study was undertaken with a view to investigate the effects of the number of bulbs on performance of A 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 θ48mm, distance from bulb to bulb 18mm,inner tube length θ 9/679mm with coil, outer tube length θ 40/720, bulbs and tubes θ 26/60mm,outlets and inlets θ 9/1.5mm and the length of quick fit 84mm.Ten (10) pieces of each type "As" and "Bs" of this modified structures of Graham condensing models were fabricated. For type "As" Graham condensers, the outlets and inlet tubes were joined to their jackets while for the type "Bs", Graham models, the outlet tubes and the inlet tubes were joined directly to their inner tubes. The performance of these condensers were evaluated using industrial grade acetone (CH_3COCH_3) for extraction of natural dye from *Lawsonia inermis* (lallebature) plant leaves, recovery of solvent and simple distillation of borehole. Results of extraction obtained shows that the highest yield was observed in condensing models with 7 bulbs in both types "As" Graham with 43.49% and type "Bs" model with 41.36% yield on extraction. The highest volume of solvent recovery was of the Graham models, with 2 bulbs on the outer jacket for both types "As" and "Bs".255mls and 234mls by volume respectively. Condensing models with 3 bulbs of Graham condenser type "As" produced the highest volume of 270mls followed by model with 9 bulbs with total distillate of 260mls then, the type "Bs" model with 10 bulbs produced the highest distillates of 283mls followed by condensing model with 3 bulbs 264mls, these distillates were collected for a period of 1 hour at intervals of 5mins each. Statistical analysis using ANOVA indicated that increase in the number of bulbs on the outer jacket glass condensers have significant effects on the overall performances on extraction processes while increase on the number of bulbs have no significant effects on the overall performance in terms of solvent recovery and simple distillation. This analogy helps in determining which kind of models to be used in performing experiments for optimum yield and minimize materials used for fabrications.

Keywords Graham Glass, Natural dye, Solvent Recovery Process

Introduction

Optimization of Graham Glass Condenser for Extraction of Natural Dye

Condenser is a heat exchanger device which uses the principle of heat transfer. A heat exchanger is a device that is used to transfer thermal energy between two or more fluids (from one medium to another) a 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].

A condenser usually consists of concentric tubes (a large glass tube containing a smaller glass tube running its entire length, within which the hot fluids pass).The ends of the inner glass are usually not fitted with ground glass joints, which are easily fitted with other glassware. During reflux, the upper end is usually left open to the atmosphere or vented through a bubbler or a drying tube to prevent the access of water or oxygen [3].

The outer glass tube usually has two hose connections, and a coolant (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 minimize the risk of thermal shock to adjacent glassware, the coolant usually enters through the lower fitting and exists through the higher fitting [4]. 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 [5].

Giancoli [6], and Ramgopal [7], stated that typical applications involve heating or cooling of a fluid stream of concern and evaporation or condensation of single or multicomponent fluid streams. In other applications, the objective may be to recover or reject heat, or sterilize, pasteurize, fractionate, distil, concentrate, crystallize, or control a process fluid. Not only are heat exchangers often used in the process, power, petroleum, transportation, air-conditioning, refrigeration, cryogenic, heat recovery, alternative fuel and manufacturing industries, they also serve as key component of many industrial products available in the marketplace [8-9].

Jensen [10], viewed Liebig, Allihn and Graham glass condensers in spite of being the most frequently used heat exchangers in teaching and research laboratories; there is still little or no improvement in terms of efficiency apart from Friedrichs and Dimroth condensers.

This research paper focuses on the laboratory Graham glass condenser. The Graham condensers consist of an inner coil surrounded by an outer tube. The coolant flows through the outer tube and substance condenses inside the inner coil. Graham condensers should not be confused with condensers. They cannot be used in refluxes either. Due to the narrow path of the inner coil, pressure rises too much and vapour start shooting out from the top of the condenser, explosion due to too much pressure could also occurred [11].

The later condensers were enhancements on Graham condensers, but are hardly obtainable or even used in most laboratories. Their low esteem is not unrelated to their difficulty, high cost of fabrication, maintenance cost and tied with the fact that they are not better off than the Graham condensing model [12].

Methodology

Design of Condensers

According to Ramgopal [7], proper design or selection of condensers are very important factors for satisfactory performance of cooling system. Conventionally, the design and lengths specification for glass condensers in practice, range from 120mm to 600mm, according to Bureau of Indian Standards (2006). AutoCAD was used to design Graham condensers varying the number of bulbs from one to ten (1-10) on the outer jacket; while keeping the lengths (720mm) constant, maintaining outer bulbs size 48mmdiameter and the distance from bulb to another 18mm with the view of increasing the surface area. Ten (10) pieces of each type “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 type “As” Graham condensers, the outlets tubes and inlet tubes were joined to their jackets while for the type “Bs” Graham condensers, the outlet tubes and the inlet tubes were joined directly to their inner tubes. The difference in design and operation of type “As” Graham condensers and type ‘Bs’ Graham condensers is the coolant flows in the outer tube for the type “As” Graham condensing models while for type “Bs”, the coolant flows in the inner tubes. Fabrication was done at Scientific Equipment Development Institute (SEDI), Minna, Niger State of Nigeria. Standard method of scientific glass technology fabrication of condensers was adopted used suitable tools [5, 13-14].

Fabrication of Graham Condensers

About 48” length of 6mm diameter glass tubing and mandrel covered with ceramic paper were 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 knitted 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 bent 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 in Allihn condensing model was employed in blowing of the bulbs on the outer jacket for the twenty assorted pieces of type “As” and “Bs” of condensing models to increase the surface area. The performance of these models was evaluated using industrial acetone grade (CH_3COCH_3) for extraction of natural dye from *Lawsoniainermis* (lallebature) plant leaves and the rate solvent recovery.

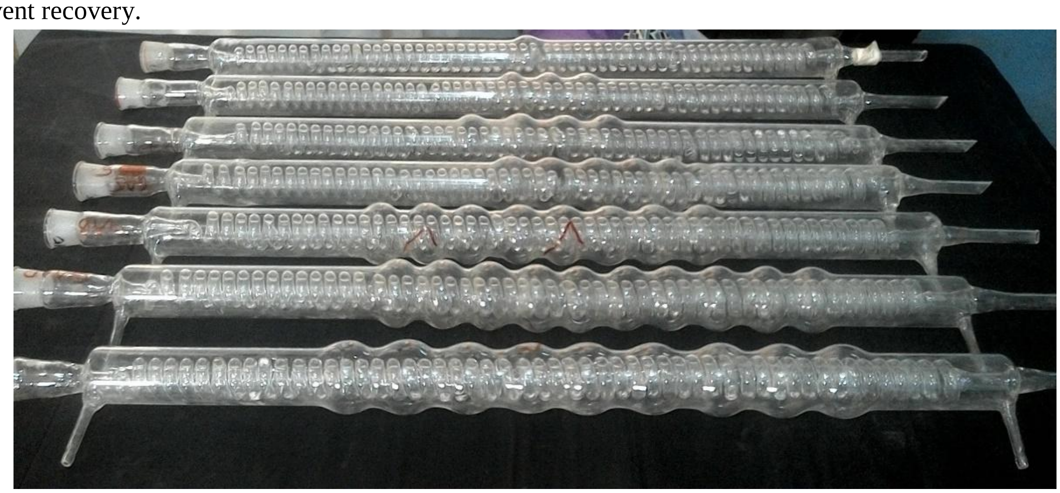

Plate I: Samples of type “As” Graham Condenser (conventional)

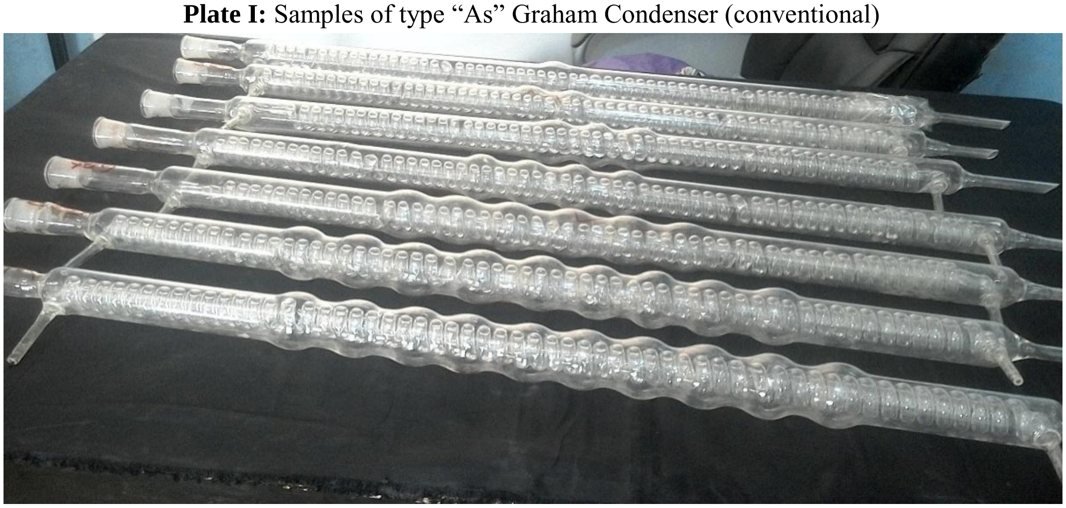

Plate II: Samples of type “Bs” Graham Condenser (unconventional)

Characterization by extraction of natural dye procedure

Graham condensers was used to carry out solvent extraction of *Lawsonia inermis* (lallebature) plant leaves. The leaves were shade dried and washed with water to removed dirt and other adhering materials, dried in a tray drier at 80˚C for 2 hours and finely powdered with the help of a grinding machine. 20g of the ground *Lawsonia inermis* (lallebature) was wrapped in a thimbles, placed inside a 500mls soxhlet extractors. The four soxhlet extractors were fitted or connected in series with500ml round bottom flasks serving as the reservoir for the concentration. 300mls of the solvent (acetone) was poured through the soxhlet opening to allow the samples and the solvent to saturate in the extracting chambers. The four sets of the condensers were connected vertically at reflux position set at 50^{o}C and operate concurrently, the temperature was allowed to gradually build up to boiling point 56^{o}C which is the b.p of solvent used. Extraction was carried out for up to 6 hours for all the samples. Acetone was used as the solvent and water as the coolant flowed through the hose connected from the water source to the inlet tube and another hose was connected to the outlet tube for the four extraction set-up. Heat was supplied from four electric heating mantles at a constant temperature but lower than the boiling point of the solvent. The mixture of solvent and grounded *Lawsonia inermis* continued to siphon into the reservoirs for six hours till the solvent in the soxhlet extractor chambers was almost colourless from the initial green colour. Thus, extraction is said to be completed when the solvent in the Soxhlet extractor is colourless [15-16]. The extracts were rinsed properly then transferred from the reservoirs into collecting plastic containers and placed on top of water bath set at a temperature below the boiling point of the solvent. This was allowed so that the remaining solvent could gradually evaporate leaving only the extract which was then, weighed using analytical balance.

Recovery Procedure

After the extraction period of six hours the thimbles containing the chaff were removed from the batch of the soxhlet extractor chambers. The heating mantles were turned on to heat the reservoir containing the extracts and solvent. The condensed solvent continued to siphoned back to the soxhlet extractor chambers till it filled up to 2/3 of each of the four extractor chambers. The solvent were then transferred into 50mls collecting measuring cylinder and the readings were recorded . The process was repeated severally untill the reservoir was partially dry leaving only the extracts, which was poured into collection plastic containers.

Results and Discussion

Results of Extraction and Solvent Recovery

Bar charts illustrate the performance of individual condenser model of both types "As" and "Bs" Grahams condensers. However, the accomplishment of this task hinge on heat energy engaged and type of heat exchanger used.

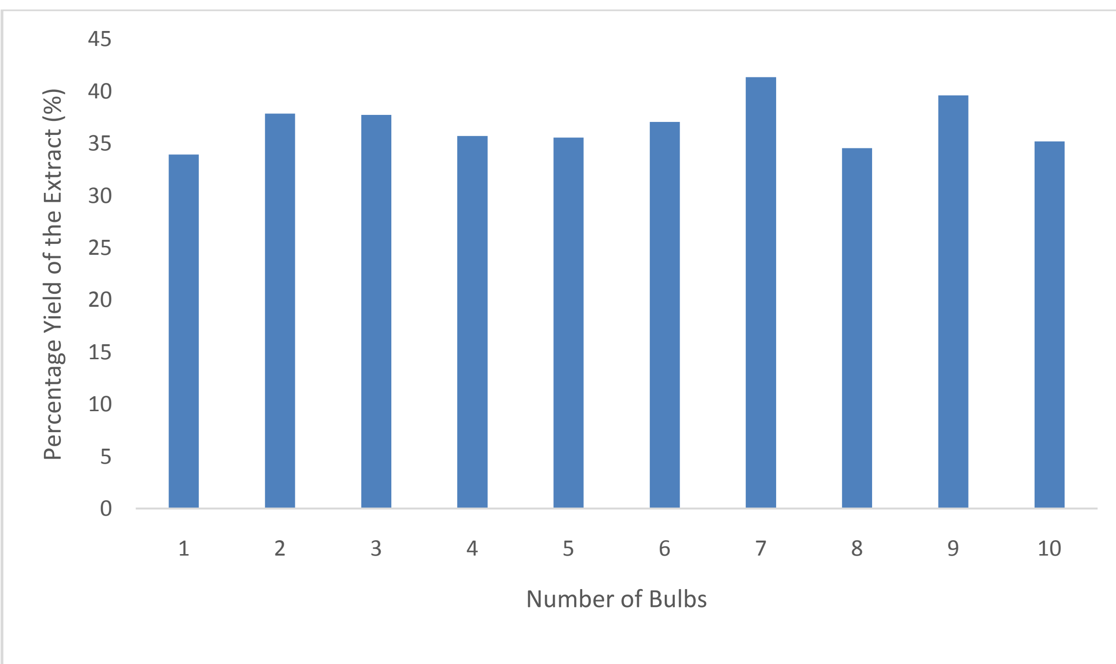

Figure 1: Percentage Yield of Type"As" Grahams Condensers

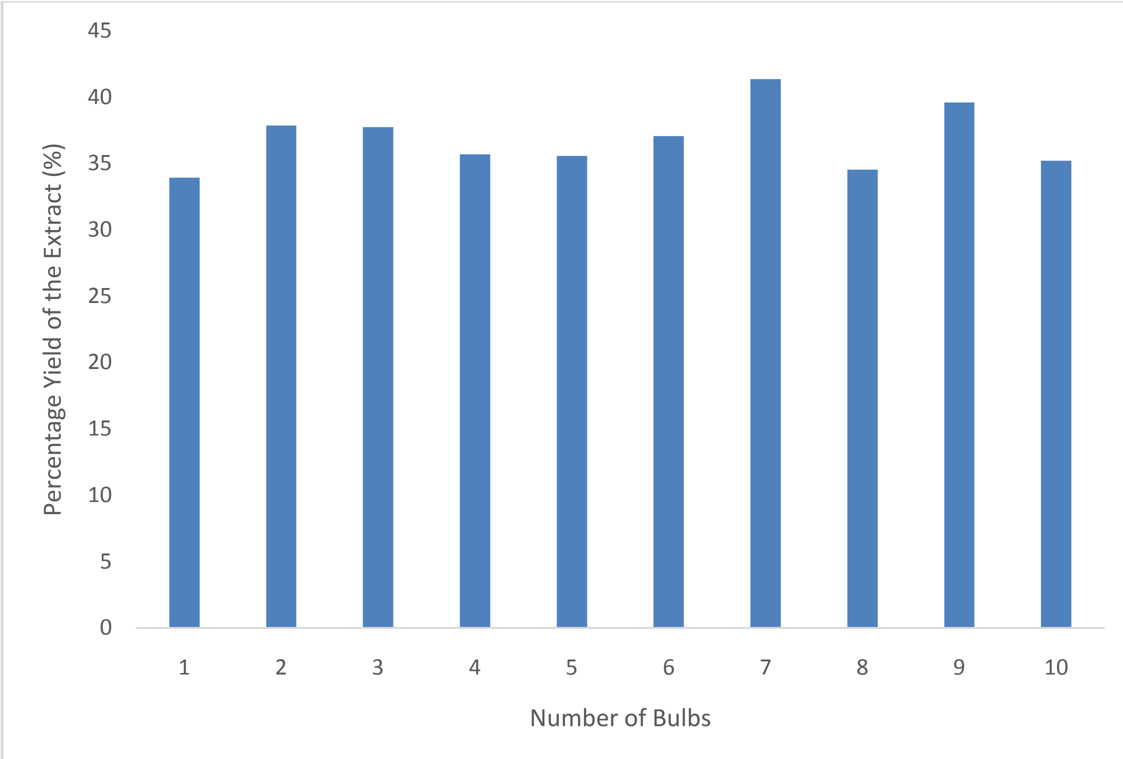

Figure 2: Percentage Yield of Type"Bs" Graham Condensers

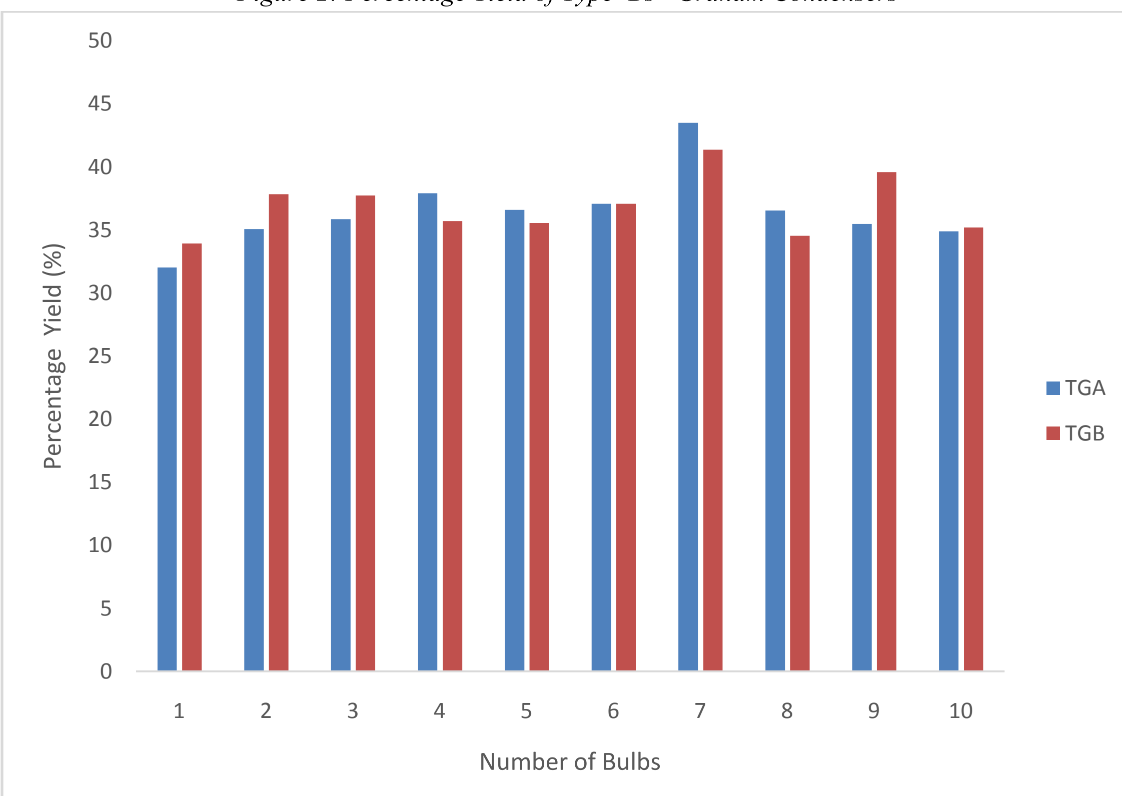

Figure 3: Percentage Yield of Types 'As" and "Bs" Graham Condensers

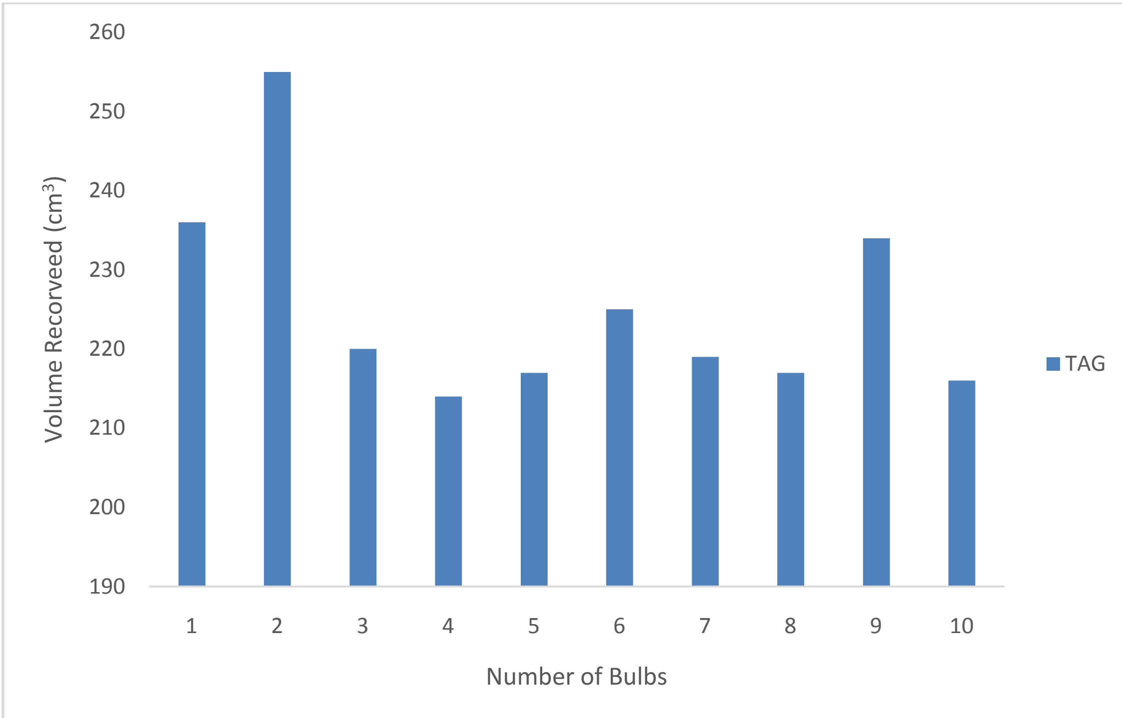

Figure 4: Solvent Recovered of with Type "As" Graham Condensers

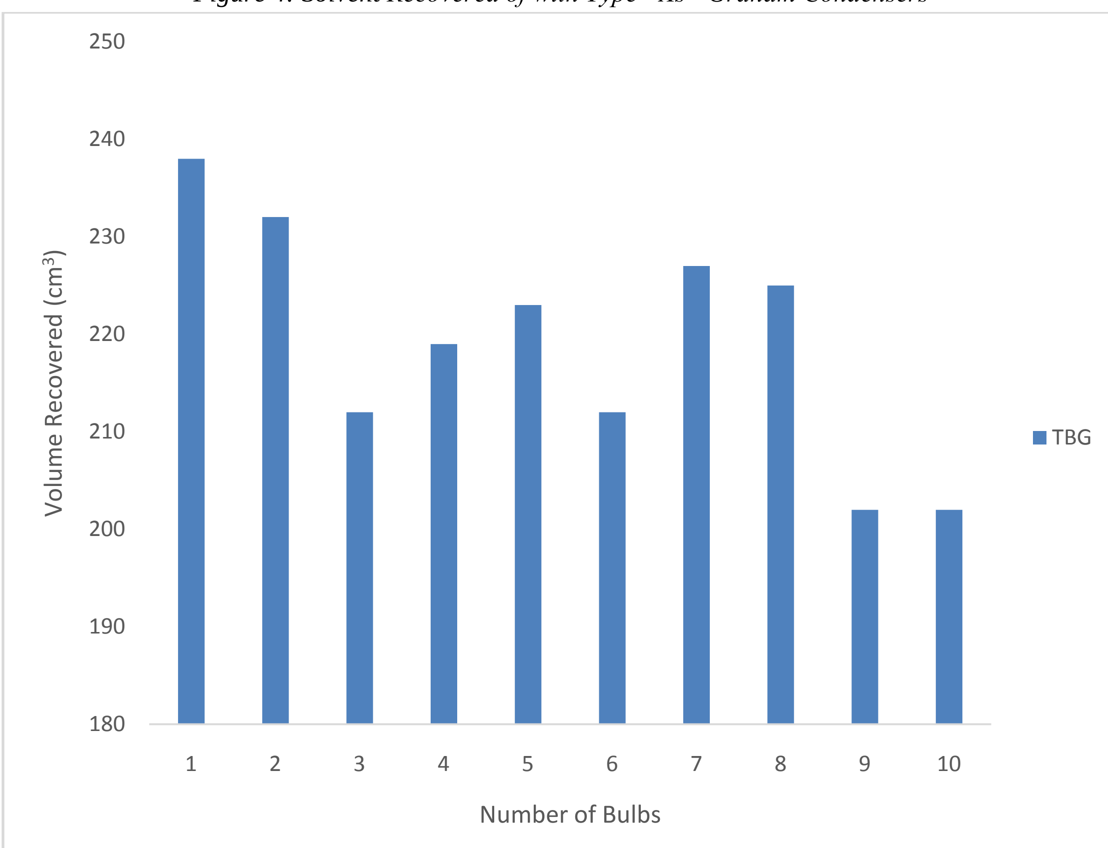

Figure 5: Solvent Recovered of with Type "Bs" Graham Condensers

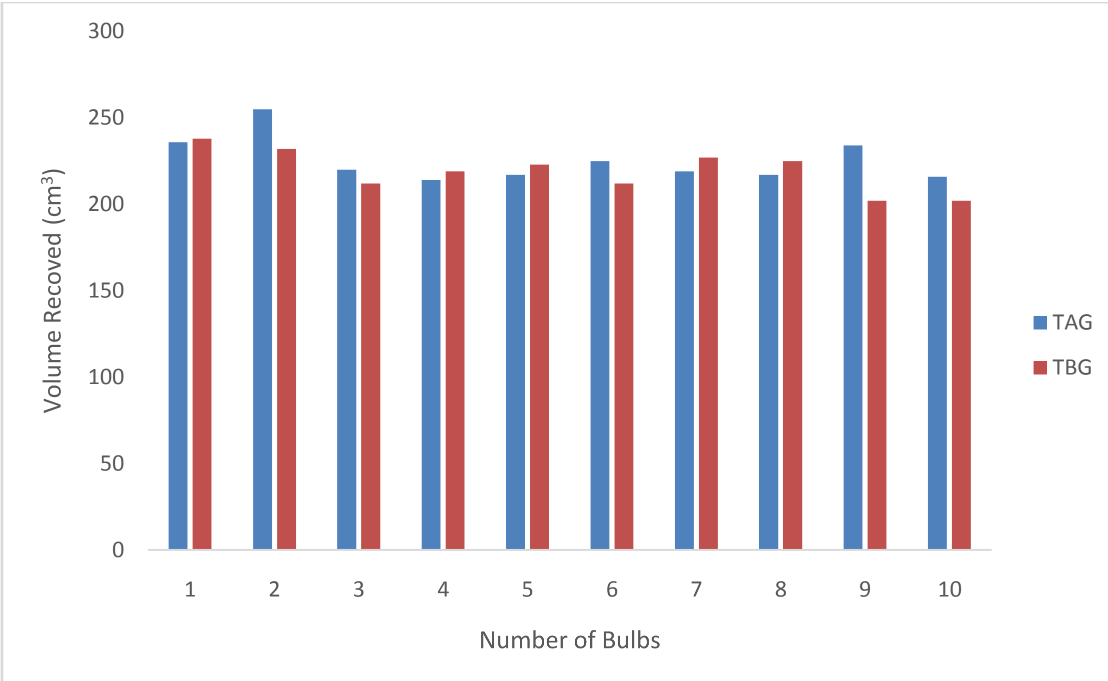

Figure 6: Solvent Recovered of with Types "As" and "Bs" Graham Condenser/s

Table 1: T-Test of Graham Types"As" and "Bs" on Extraction

Percentage yield of extracts	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
"As"	10	37.00	2.47	18	0.131	0.897	Retain Ho
"Bs"	10	36.86	2.34				

Table 2: T-Test of Graham Types-"As" and "Bs" Solvent Recovery

Final vol. of solvent (mls)	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
"As"	10	225.30	12.88	18	1.091	0.290	RetainHo
"Bs"	10	219.20	12.12				

Graham condenser models, type "As'' from Figure 1 show that condensing model with 7 bulbs produced 43.49% extract while the lowest percentage of extract with 32.03% was observed in condensing model with 1 bulb. These abnormal behaviour observed in condensing model with one bulb is likely as a result of the prolonged contact with the coolant owing to the fact that the entire length is spiral throughout in nature, which reduces the effectiveness of the heat changers in term of surface area. As indicated in Figure 2 of Graham type "Bs" condenser with 7 bulbs also produced the highest percentage of extracts 41.36%.Graham condenser with 7 bulbs all had the highest percentage of extract obtained. As observed in figure 3 both Graham types "As" and "Bs" shows that types "Bs" and "As" Graham condensing models have more percentage yields of extracts. Figure 4 shows the overall performance of the models which established the effectiveness and optimum extraction was found on the condensing models with numbers of 7 bulbs with the highest yield of extracts. Further fabricating of this condensing models with bulbs above or below 7 will amount to waste of production, cost, materials and time. This shows with the improvement in surface area in both types of models could be used in reflux position as against the backdrop that are not suitable in reflux position (Bon, 2010).

Recovery of Solvent

Figure 5 shows Graham types "As" condensing model with 2 bulbs gave the highest yield of the solvent recovered with 255mls followed by the model with 1 bulb 236mls and the lowest recovery was 214mls from condensing model with 4 bulbs. As presented in Figure 6 recovery of solvent with type "Bs" Graham condensing models with 1 bulb and 2 bulbs produced the highest volume of 234mls and 232mls respectively. Lowest solvent recovery was 202mls from condensing models with 9 and 10 bulbs. Figure 7 shows recovery of solvent with types "As" and "Bs" Graham condensers. Models with two bulbs have the highest solvent recovered with 487mls while the least of 418mls was in condensing model with 10 bulbs. This suggest that the optimum recovery rate was found in condensing models with 2 bulbs and it should be the preferred condensing models because it is very effective, less materials will be require for fabricating it, cost effectiveness, better yield and consequently time saving.

Statistical Analyses

Statistical analysis was used to establish the accuracy of the results collected from the evaluation of condensers used for extraction of natural dye, solvent recovery and simple distillation of borehole water. T –test was used by sample pairing to compare the forty pieces of types "As" and "Bs" of Graham, based on the effects of the number of bulbs on the performance. Analysis of variance (ANOVA) was also used to compare the individual effects of the number of bulbs on performance of Graham types "As" and "Bs" glass condensing models.

In Table 1 and 2 Graham condensing models of types "As" and "Bs" presented the same trend as above where by the sample paired T-test value was lower than T-critical and that of the p-value, greater than α value at 0.01 level of significant. Therefore Ho is retained, this showed that there is no significant difference on the overall performance of the models regardless the number of bulbs on the outer jacket of the condensing models of Graham.

Conclusion

Assorted condensing models with 7 bulbs on the outer jacket are generally more suitable and efficient for extraction with Graham condensing models types "As" and "Bs". Due to increase in surface area. Graham type "As" with 2 bulbs condensing models are very effective in terms of solvent recovery. From the forgoing it is enlightens the scientific glass technologist and the end users in selecting the ideal design, length, cost saving and the glass materials to be used for the fabrications.

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