Optimization of Allihn Condensers for Solvent Extraction of Neem Oil for Industrial Application

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ABSTRACT

The effect of the number of bulbs on the contact time for solvent extraction of oil from Neem seed was studied. Ten type "As" (A1 to A10) and ten type "Bs" (B1 to B10) Allihn condenser models were designed using autoCAD software and fabricated with borosilicate glass. The same dimension of inner tube lengths θ9/556mm, outer tube lengths θ40/600mm, outlets and inlets θ9/1.5mm and diameter of the bulbs θ26/50mm were maintained. Time to complete extraction, neem oil extract, solvent recovered, residue, number of siphon and pH of the extract were the parameters considered. 200ml of analar grade ethanol, temperature of 50°C, and 20g ground neem seed were used for this experiment. An average extract of 4.80g for type "As" Allihn condensers and 5.99g for type "Bs" Allihn condensers were recorded. Type "As" retained an average of 122.99ml solvent with type "Bs" 119.80ml but type B8 (147.76ml) retained the most solvent. Type "Bs" had the least average residue of 12.61g compared to 12.95g of type "As". Type "Bs" had an average of 18.20 siphon higher than 15.20 of type "As". pH of the extract was 6.5 which falls within the range of 5.7 - 6.5 standard value which can be applicable for natural pesticides, insecticides, insect repellants and fertilizers. B3 (10.79g) gave the highest yield of neem oil in 3hrs 30mins but for optimum result, type "Bs" Allihn condensers should be used. Emphasis on type B3, B4, B5 or B6 can be used in the process saving cost and time.

Keywords*:* Neem seed, Neem oil, Allihn condensers, Solvent extraction and Optimization

INTRODUCTION

Condensers are heat exchangers which operate by removing heat from gas or vapour; once sufficient heat is eliminated, liquefaction occurs. For some applications, all that is necessary is to pass the gas through a long tube (usually arranged in a coil or other compact shape) to permit heat to escape into the surrounding air (Som, 2008 and Mansur *et al.,* 2016). The system is in steady state until conditions change, such as flow rate or inlet temperature. These changes in conditions cause the temperature distribution to change with time until a new steady state is reached. The new steady state will be observed once the inlet and outlet temperatures for the process and coolant fluid become stable. In reality, the temperatures will never be completely stable, but with large enough changes in inlet temperatures or flow rates a relative steady state can be experimentally observed (Jeffrey, 2002).

Today's heat exchangers must meet a variety of highly demanding requirements. In terms of performance, they have to ensure maximum heat transfer while keeping size to a minimum. Furthermore, the durability of heat exchangers must be extremely high, providing trouble-free performance throughout its service life at low manufacturing costs (Parag and Manoj, 2015). The varying demands of heat exchangers in various chemical processes using condensers have therefore led to the design of varieties of types (Liebig, Allihn, Graham and zigzag condensers). A general design theme being creative ways in which the surface area for vapour-liquid interaction and heat exchange can be increased, a standard related to an apparatus's efficiency in separating components with smaller differences in boiling point, and ways in which to control common difficulties experienced in real distillations (Armarego and Chai, 2012).

Allihn condensers are glass laboratory condensers designed to improve on the existing condensers by having a wider bore at the bottom and condensing the liquid on the bubbles (increasing the surface area for contact with the liquid) where it can run down the sides and avoid blocking the vapour. A lot of them come with very wide male sockets on the base to fit the equally wide top of a Soxhlet extractor; which needs to be wide to load the thimble (Bon, 2010). Allihn coolers provide about 30% higher cooling capacity than an equivalent Liebig. They are ideal for vertical condenser jobs like total reflux or extraction using Soxhlet extractor (Gert, 2012).

Scientific Glass Technology (SGT) is a basic industry within the larger scientific community and glassblowers focus on designing, constructing, modifying, repairing and maintaining laboratory glass instruments (Gonah, 2014). The borosilicate glass family is the most important to scientific glass blowers and chemical apparatus because of its very good chemical and physical properties. It is considered technically as all round glass for applications requiring very good chemical and thermal resistance (including resistance to thermal shock), and high mechanical stability. Typical applications are as components for chemical apparatus, round-bottom flasks, and beakers (LABSOLV, 2015).

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 is an important part in the process of development and production of a desired product. For production on an industrial scale, minimal equipments of glassware which would be used in the industries have to be made (Braz, 2012). Moreover, laboratory glass wares, which are the bedrock of scientific glass instruments, especially heat exchangers do not receive the desired attention indigenously as most of them are still imported (Gonah, 2014).

Solvent extraction, mechanical pressing and supercritical fluid extraction are some of the methods used to obtain Neem oil from the seed. Mechanical extraction is the most widely used method to extract Neem oil from Neem seed. However, the oil produced with this method usually has a low price value, since it is turbid and contains a significant amount of water and metal. The oil produced from supercritical fluid extraction, has very high purity; however the operating and investment cost is high. Extraction using solvent has several advantages; it gives higher yield, less turbid than oil obtained from mechanical extraction and also has low operating cost (Liaw *et al.*, 2008). Another importance of solvent extraction is that the solvent can be reused. Ideally in a close system like extraction, the amount of solvent used should be the same recovered. However, it is not so in real life situations due to losses incurred during handling before and after extraction.

The bulbs in Allihn condensers are the reason for their efficiency in refluxing, less susceptibility to clogging, 30% better than Liebig condensers and large surface area available for the interaction of the solvent with the materials (Gert, 2012). However, the above qualities neither give any idea of the size of the bulbs nor the number of bulbs per standard condenser length of 120mm, 240mm, 360mm, 480mm and 600mm. This study therefore investigated the effects of bulbs on the performance of Allihn types "As" and "Bs" condensers.

METHODOLOGY

Twenty Allihn condensers, ten type "As" and ten type "Bs" Allihn condensers of length 600mm were designed and fabricated. Fabrication was done by heating a portion of a borosilicate glass tube until it was plastic enough to be shaped by bending, blowing, pulling, pushing and tooling. Basic operations that were used to fabricate the various glass systems of the twenty Allihn condensers were pulling off points (pulling off spindles or drawing peaks or pitz), fire-polishing of tube ends, flaring of tube openings, tubes bending, T-seals and blowing bulbs at Scientific Equipment Development Institute (SEDI), Minna, Niger state. The efficiency of the twenty condensers were tested by extraction of Neem oil from ground Neem seed at National Research Institute for Chemical Technology, Zaria.

Designing of Allihn Condenser

AutoCAD is one of the most important designing and drafting tool hence, was used to design the Allihn condensers varying the number of bulbs from one to ten while maintaining the same dimensions of inner tube lengths θ9/556mm, outer tube lengths θ40/600mm, outlets and inlets θ9/1.5mm and diameter of the bulbs θ26/50mm. The difference in design and operation of type "As" Allihn condensers and type "Bs" Allihn condensers is the coolant flows in the outer tube for the type "As" Allihn condensers while for type "Bs", the coolant flows in the inner bulbs.

Design models of Allihn condensers

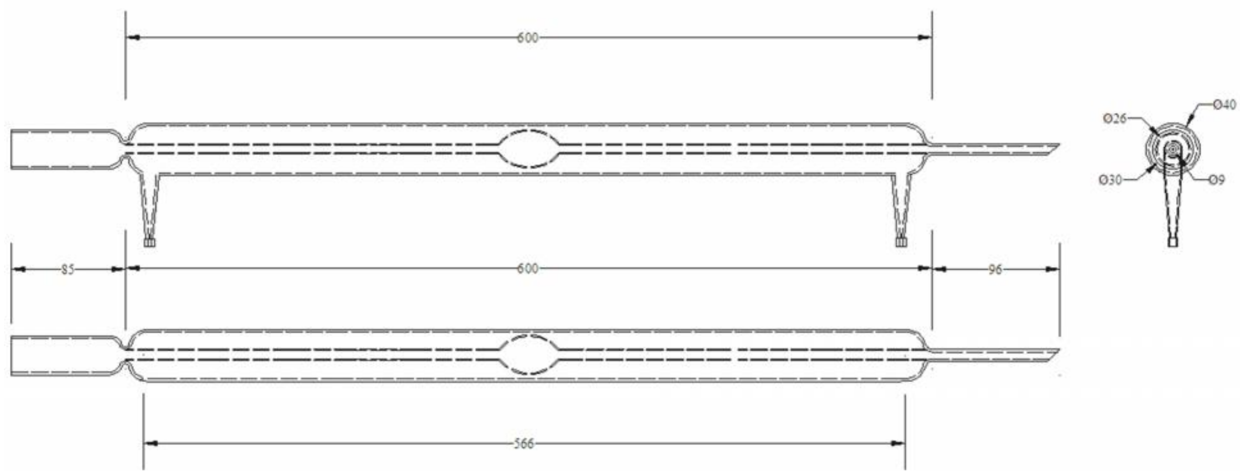

Figure 1: Type A1 Allihn Condenser (Conventional)

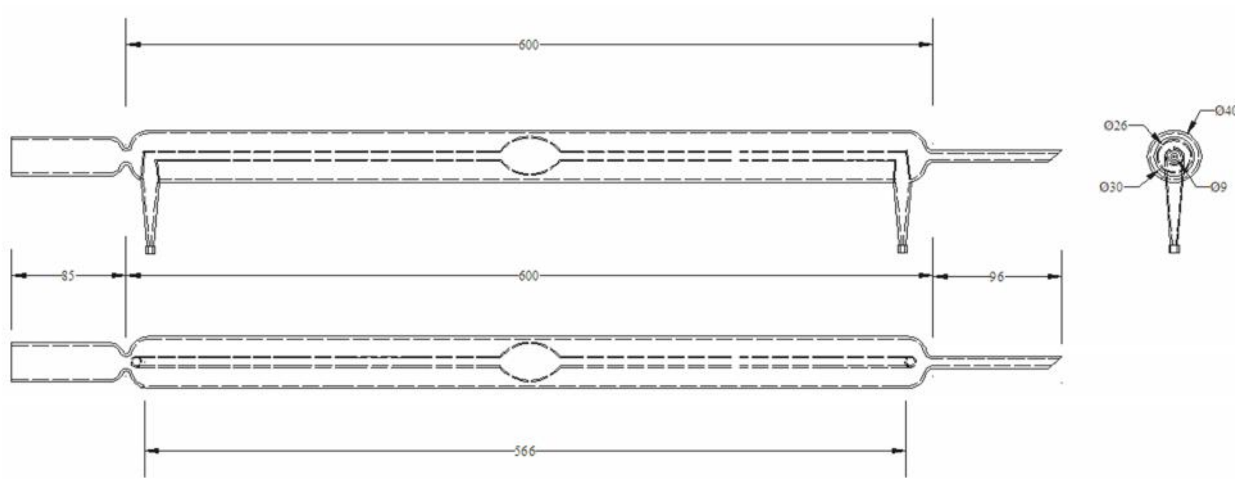

Figure 2: Type B1 Allihn Condenser (Unconventional)

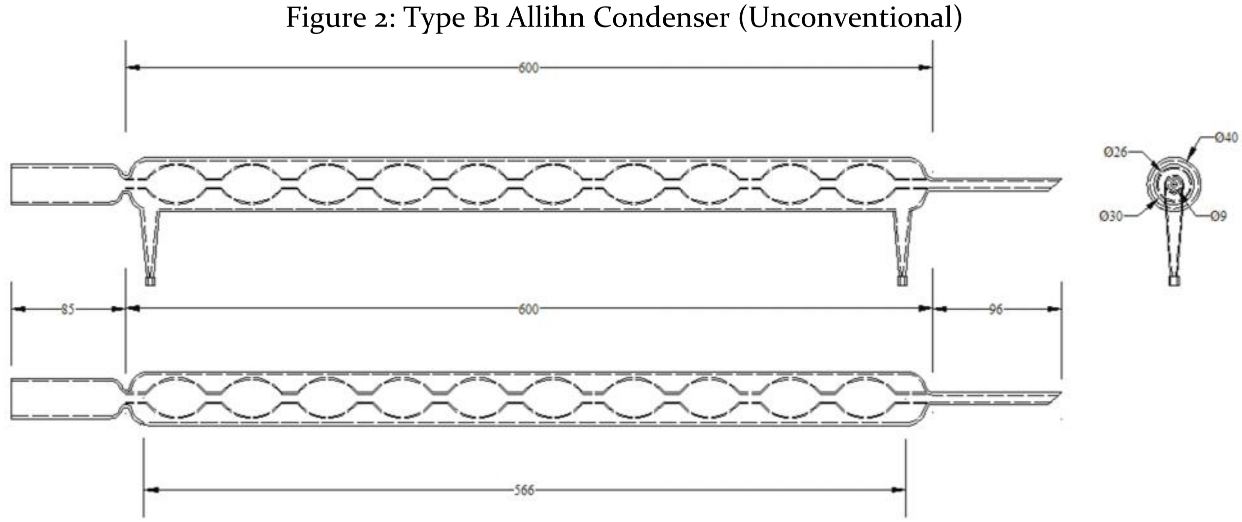

Figure 3: Type A10 Allihn Condenser (Conventional)

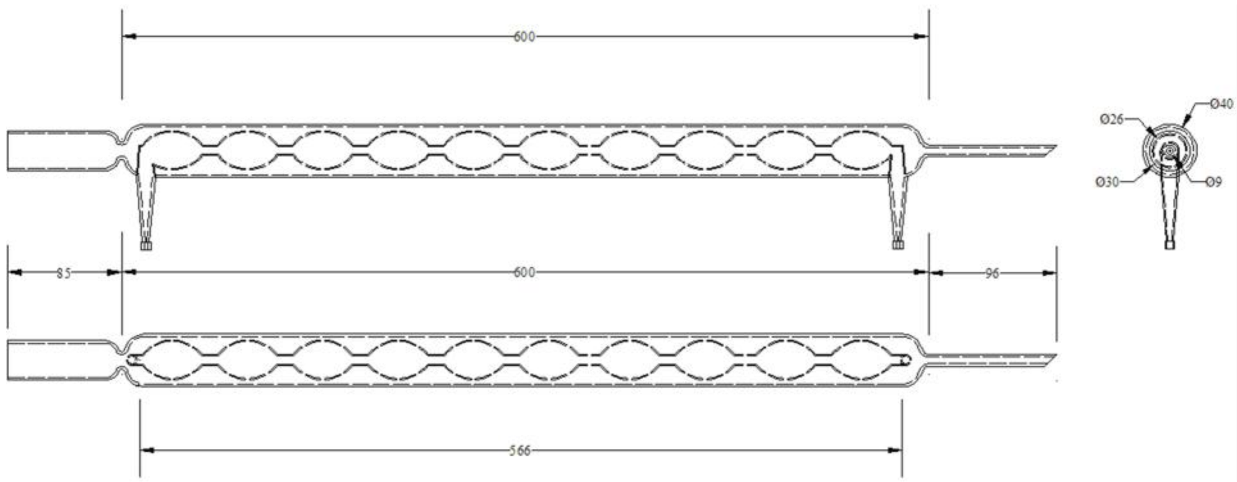

Figure 4: Type B10 Allihn Condenser (Unconventional)

Characterization of Allihn Condensers

The efficiency of the fabricated Allihn condensers was tested by carrying out solvent extraction of Neem oil from ground Neem seed. 20g of ground neem was weighed and poured into a filter paper, inserted into a Soxhlet apparatus fitted with a 250ml capacity round bottom flask containing 200ml analaR ethanol. The Allihn condenser was fitted to the Soxhlet apparatus, placed on the heating mantle and clamped. Rubber hose were connected to the inlet and outlet tubes of the condenser and the tap turned on for the coolant (water) to flow.

For proper extraction to take place and to obtain the highest quality of oil, the heating mantles were set at 50^{o}C (Liauw *et al*. 2008). At the first siphon, a golden yellow colour fluid was observed. This showed that interaction between the solvent and neem seed had begun. As the siphoning of the solvent and neem oil continued, the colour grew darker until it turned brown because more neem oil was extracted. After a while, the mixture in the Soxhlet extractor started to clear as the neem oil in the Soxhlet extractor decreased until it became completely clear. Both the time for complete extraction and number of siphon was noted at the end of each extraction before turning off the heating mantle. The mixture of neem oil and solvent was poured into a labeled 200ml volumetric flask. Extraction time for all the twenty Allihn condensers lasted between three hours three minutes (3hrs 3mins) to six hours thirty five minutes (6hrs 25mins).

To separate the mixture of the neem oil and solvent, a SENCO rotary evaporator was used. The neem oil was stored in labelled sample bottles and the residue from each extraction was reweighed. The pH of the extract was tested using a pocket pH meter.

RESULTS AND DISCUSSION

Results for Extraction

The results of the efficiency of the designed and fabricated twenty Allihn condensers by solvent extraction of neem oil from neem seed are reported based on: time to complete extraction, extract, residue, solvent recovered and pH of the extract were the parameters considered.

Time for complete extraction

Figure 5 shows type "As" and type "Bs" Allihn condensers took an average time of 4hrs 14mins and 4hrs 22mins respectively to complete extraction. Types B8 (3hrs 3mins) and A9 (6hrs 35mins) had the least and most time to complete extraction respectively. This shows that type "As" Allihn condensers completed extraction in a shorter time compared to type "Bs" Allihn condensers.

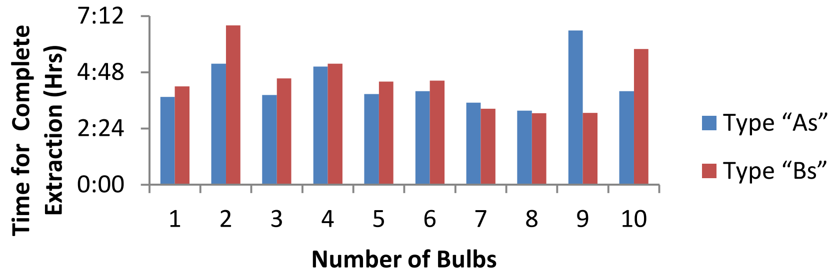

Figure 5: Time Taken for Complete Extraction of Oil from Neem Seed Using Allihn Conventional (Type "As") and Unconventional (Type "Bs") Condensers

Extract (neem oil)

Type "As" gave an average extracts of 4.80g and 5.99g for type "Bs" Allihn condensers. Types B3 (10.79g) and B8 (3.84g) had the highest and lowest amount of extract respectively. Therefore, type "Bs" Allihn condensers are more efficient in extracting Neem oil than type "As" Allihn condensers.

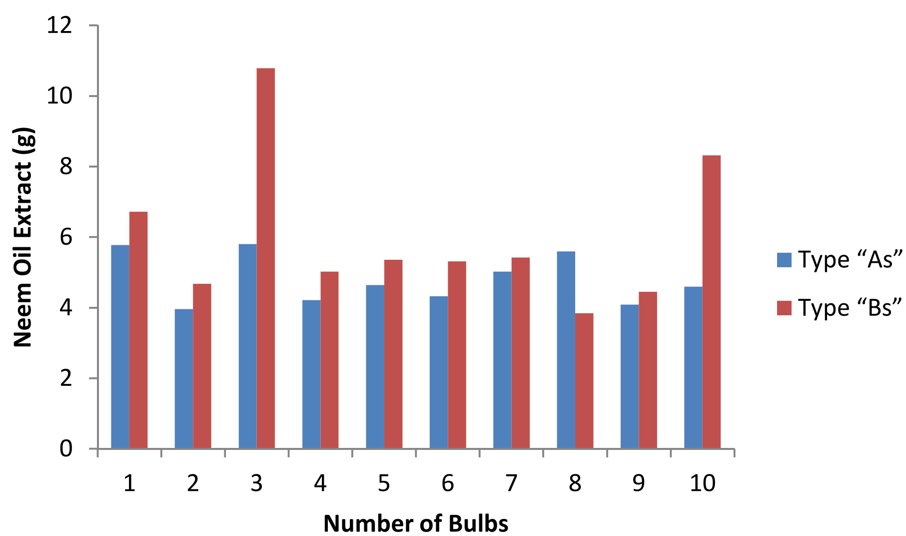

Figure 6: Weight of Neem Oil Extract from Allihn Conventional (Type "As") and Unconventional (Type "Bs") Condensers

Solvent recovered

Type "As" retained an average of 122.99ml solvent with type "Bs" 119.80ml but type B8 (147.76ml) retained the most solvent. Type "Bs" Allihn condensers had both the most B8 (147.76ml) and least B1 (103.30ml) solvent recovered. This shows that with increase in the number of bulbs, type "Bs" Allihn condensers minimized loss of solvent during extraction.

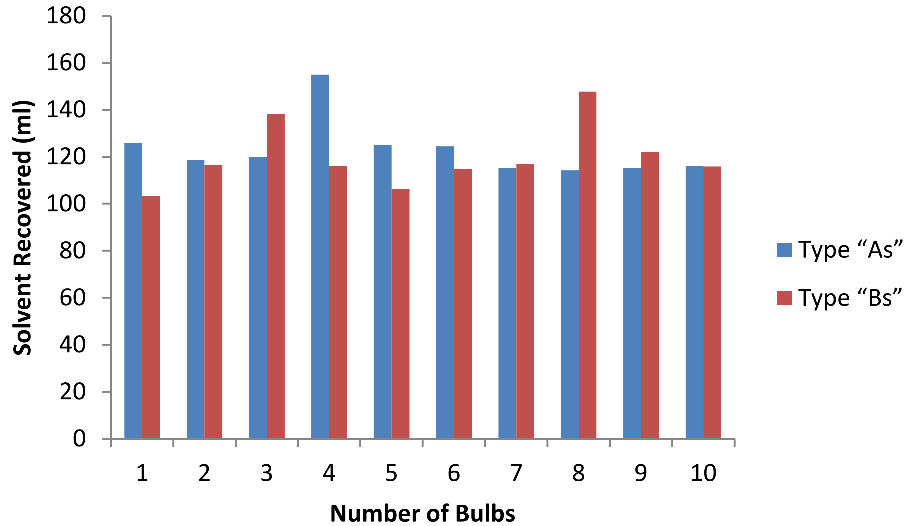

Figure 7: Solvent recovered from Allihn Conventional (Type "As") and Unconventional (Type "Bs") Condensers

Residue

Type "Bs" had the least average residue of 12.61g compared to 12.95g of type "As". Types A10 (15.05g) and B3 (11.95g) had the highest and lowest residue respectively. This shows that type "As" Allihn condensers extracted less oil than type "Bs" Allihn condensers. The higher the Neem oil extracted, the less residue.

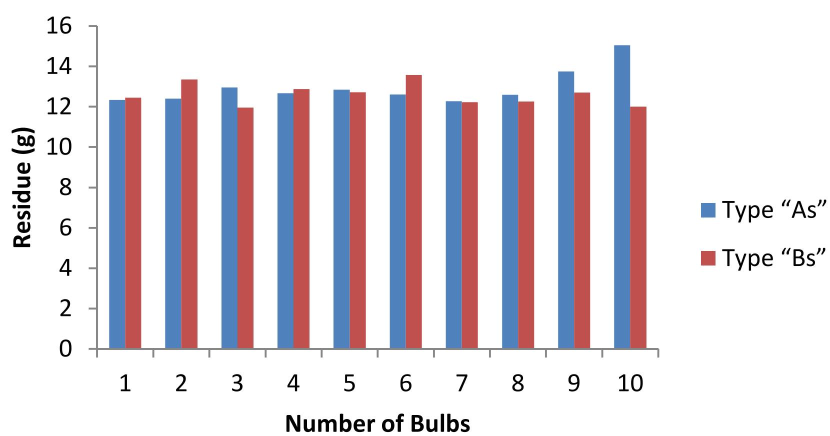

Figure 8: Weight of Residue of Neem Seed after Extraction for Allihn Conventional (Type "As") and Unconventional (Type "Bs") Condensers

Number of siphon to complete extraction

From Figure 9 types B2 (26) and A6 (12) had the highest and lowest number of siphon to complete the extraction. Type "Bs" had an average of 18.20 siphon higher than 15.20 of type "As". This shows type "Bs" Allihn condensers cooled the system more efficiently than type "As" Allihn condensers.

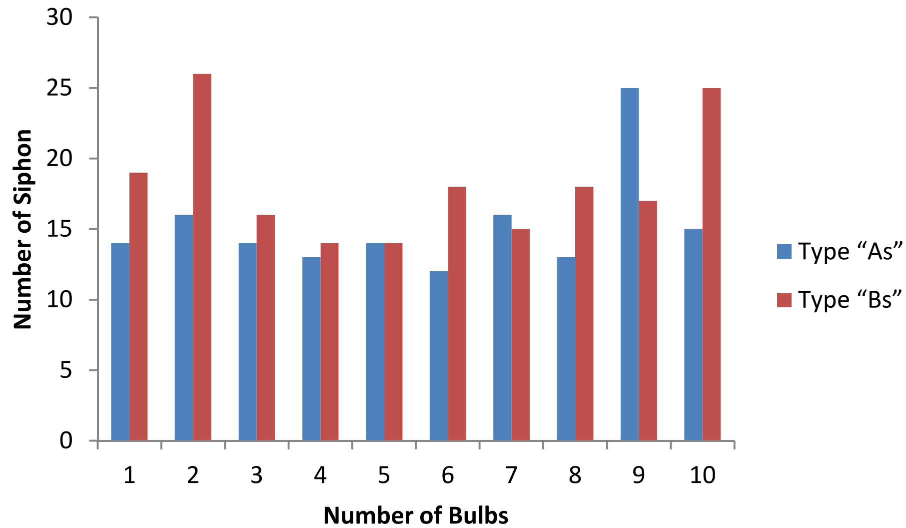

Figure 9: Graph of Number of Siphon for Types "As" and "Bs" Allihn Condensers

pH of the Neem Oil (Extract)

The pH of the extract was 6.5 and was constant for all the types "As" and "Bs" Allihn condensers. The pH falls within the range of 5.7 - 6.5 standard value (Liauw *et al*. 2008). This shows that both Type "As" conventional and type "Bs" unconventional Allihn condensers extracted Neem Oil but type "Bs" Allihn condensers are more efficient.

CONCLUSION

Based on the results of this study type B3 Allihn condenser with three number of bulbs gave the highest yield of 10.79g in 3hrs 30mins. Type "Bs" which is the unconventional Allihn condensers gave an average yield of 5.99g which is higher than 4.80g for type "As" conventional Allihn condensers. Therefore, type "Bs" unconventional Allihn condensers are more efficient for solvent extraction. However, type "Bs" Allihn condensers with three bulbs, four bulbs, five bulbs and six bulbs give optimum yield hereby saving time and cost.

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