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Optimization of Methanol-Water Extraction of Plant Materials for the Production of Coloring matter

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Abstract
Great emphasis is put in the retrieval of natural dyes which are nowadays gaining popularity due to their green approach. Three different plants were selected in this study. A comparison was made between two different extraction techniques which are the microwave oven and magnetic stirrer. The results obtained demonstrated that the extraction yield using microwave technique was higher than that of magnetic stirrer for all the three different selected plants for the study. The experiments were conducted using different SSR of 1/15, 1/20 and 1/25, at different agitation speeds (100rpm, 150rpm and 200rpm), microwave oven powers (low, medium and high), and solvent composition (50% methanol 50% water, 30% methanol 70% water and 70% methanol 30% water) at selected particle size of 0.45mm

Keywords *Magnetic Stirrer, Microwave, Agitation Speed, solid: solvent, Methanol-Water, Power, Lawsonia Inermis, Cochspermum tinctorium, Parkia Biglobosa*

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I. Introduction

In the current world in which sustainability and concern about the industrial residues are emerging as a concept, there is a tendency toward a cleaner industry with better use of energy replacing whenever possible synthetic chemicals for raw materials derived from renewable resources which should be used in a rational manner so that they can be preserved in nature.

Green Chemistry is the design, development, and implementation of chemical products and processes to reduce or eliminate the use and generation of substances hazardous to human health and the environment (Manley *et al.,* 2008). According to this concept, unlike regulatory requirements for pollution prevention, Green Chemistry is an innovative, non-regulatory, economically driven approach toward sustainability. For example, textile processing industry is one of the major environmental polluters. In order to process a ton of textile about 230-270 tons of water is spent by generating an effluent proportional to this quantity (Mirjalili et *al.,* 2011; Gulrajani,). According to these data, there are two main ways to limit the environmental impact of textile processing. One is to construct sufficiently large and highly effective effluent treatment plants, and the other way is to make use of dyes and chemicals that are environment friendly (Vankar 2006; Sivakumar, 2011). Natural dyes appear to be the most appropriate substitute to the relatively toxic synthetic dyes (Patron, 1998; Norton, 1998). They are believed to be safe because of non-toxic, non-carcinogenic and biodegradable nature. Further, natural dyes do not cause pollution and waste water problems (Ali et *al.,* 2009). Natural dyes are not an innovation but a revival of a rich and prudent tradition and cannot be compared with synthetic dyes only in terms of efficiency for industrial applications. However, we need to ensure that the replacement of synthetic dyes by natural dyes will not just transform the industrial fabric into handcrafted fabric, but lead to a more environmentally friendly fabric with the same quality standards previously achieved with synthetic dyes (Guesmi et *al.,* 2012).

Therefore there is an increasing demand for developing appropriate techniques for a more efficient and effective extraction of active substances from the plants and minerals in order to enable the use large scale of natural dyes (Siva Kumar *et al.,* 2009). From the point of view of some authors (Piccoglia etal, 1998; Guesmi *et al.,* 2013) for the reintroduction of natural dyes in industrial scale, the dyeing process with natural dye should be possible to be performed on the equipment available in modern textile industry without requiring large investments.

In this context, the work aims to examine new approach for the extraction of natural dyes by varying solvent composition, solid/solvent ratio(SSR),agitation speed using magnetic stirrer and microwave extraction techniques.

II. Experimental Methods

Sampling

The dye plant materials were procured. The barks, leaves and roots of the plant materials were washed with distilled water to remove dusts and solids and then dried in air and stored in a cool place to avoid degradation of pigment prior to extraction. Using different kitchen graft slot dimensions, the dried plant parts were grinded and sieved with average particle size of 0.45mm. The reduced material was stored in a container, labeled and sealed.

Identification

The plant materials used in this work are Lawsonia Inermis (Lalle), a flowering plant and the sole species of the Lawsonia genus; it belongs to Lythraceae family, kingdom plantae. It is also called henna tree; Cochlospermum Tinctorium (Rawaya) is found in dry savannah and belongs to Cochlospermaceae, kingdom plantae; Parkia Biglobosa (Makuba) also known as Locust bean, is a perennial deciduous plant that belongs to Fabaceae family, kingdom plantae. The plants were identified by the Department of Biology, Umaru Musa Yar'adua University Nigeria.

Optimization

Three parameters were investigated to optimize the extraction yields of the colorants. These parameters were varied one at a time to identify the optimum conditions for the experimental runs.

Table1 Set of values used for experimental runs

S/N	Parameter	Selected values
1	Methanol – Water (Solvent)	30/70, 50/50, 70/30.
2	Magnetic stirrer speed (rpm)	100, 150, 200
3	Particle size of plant	0.45mm
4	Solid – solvent ration	1/15, 1/20, 1/25
5	Microwave power	Low, Medium and High powers

Magnetic Stirrer Extraction Experiment

To maximize the dye extraction yield by magnetic stirrer, three agitation speeds were used at different solvent composition, room temperature for 1hr.The average particle size of 0.45mm and solid solvent ratio (SSR) 1/15, 1/20, 1/25 were maintained.

Finely grated plant part dimensions were weighed (6.667g, 5g, 4g) respectively and put in a beaker (300ml) containing a total volume of liquid mixture (100ml) (50ml methanol; 50ml water). The beaker were covered with aluminum foil to avert loss of solvent and then magnetically stirred for a maximum of one hour. The speed of agitation was maintained at 100rpm. The experiments were repeated at 150 and 200rpm for each set of solvent mixture and solid solvent ratio (SSR).After the extraction, the extract is filtered and samples were taken and the absorbance measured spectrometric ally. The percentage extraction yields are computed from the absorbance measurements. After screening through a nylon sieve, filtered extract samples were taken at 10 min interval and the absorbance was analyzed spectrometric ally. Eventually, the % extraction yields were computed.

Experimental procedure for Microwave Oven

Microwave extraction was performed in an experimentally closed microwave oven (Trust Model, Sky run WMO 20L-MGSB) with a power of 800 W. Typically, a glass beaker (300ml) representing the extraction vessel constitutes a total solvent volume (100ml) (50 ml distilled water and 50 ml methanol). Weighted amounts (4 g – 5 g – 6.667 g) of the four grated plant equivalent to the selected SSR (1/15 – 1/20 – 1/25) were then subjected to extraction by pouring the solid matrices in the beaker. The glass beaker was situated in the center of the microwave oven, containing a circular 360° rotating carousel with several durations of exposure at a constant microwave power of low power. The beaker was covered with a watch glass to prevent loss of solvent by evaporation. For time duration of only 5 min, dye extracts were retrieved at 50 s time intervals, just after the mixture in the glass beaker was permitted to cool down to room temperature and filtered through a nylon sieve. The experiment was repeated at medium and high powers. Ultimately, using the spectrophotometer, the extract was examined by measuring the absorbance at the optimum wavelength from which the % dye extraction yield could be evaluated.

Determination of Optimum Wavelength.

The grind sieved plant material (30g), of particle size of 0.45mm was placed in a beaker (300ml) containing methanol (300ml) and stored in a refrigerator for 3 days. The concentrated dye solution is then diluted 4, 6, 8, and 10 times. Each of the diluted solution (10ml) was then dispersed into separate clean dried spectrometer glass tube and positioned into spectrometer to measure absorbance. A tube containing distilled water is inserted in the machine for zeroing the machine prior to insertion of each tube. The absorbance of the dilute solutions for each colorant is read between 400 – 650nm. The wavelength corresponding to highest absorbance value is recorded. A graph of absorbance against wavelength is plotted from which optimum wavelength for each of the dye is observed.

Preparation of Standard Curve.

The standard curve is plotted from values of absorbance of dilute solutions at pre-observed optimum wavelength for each dye material extract. The concentration of extracts from different solvent composition, solid solvent ratio, particle size and time are determined by taking absorbance values of extracts at intervals and extrapolated from the curve. The concentration of dye present in the extract is calculated based on the calibration plot after noting the absorbance. The ratio of natural dye extraction yield is then calculated using the equation of Zhang and Lui

(2008). Parentage Dye Extraction = $\frac{C_t \times V \times 100}{W_o}$

Where C_t = dye concentration in solvent at time t, mg/L V = total volume of solvent, ml.

W_o = weight of total dye present in solid plant matrices, mg

III. Results And Discussion

Determination of Standard Curve

The standard curve is plotted from values of absorbance of diluted solutions at pre-observed optimum wavelength. The concentration of extracts for various plant material, using different agitation speed and microwave power were obtained from the curves by extrapolating the results obtained.

Effect of Agitation Speed on the Extraction of the dye from plant matrices

The results of the effect of agitation speed on the extraction of the dye from Lawsonia inermis are given in Figures 1-3 . The highest yield was 70% at 150rpm and SSR 1/25 and the minimum is 23% at 100rpm and SSR 1/15.

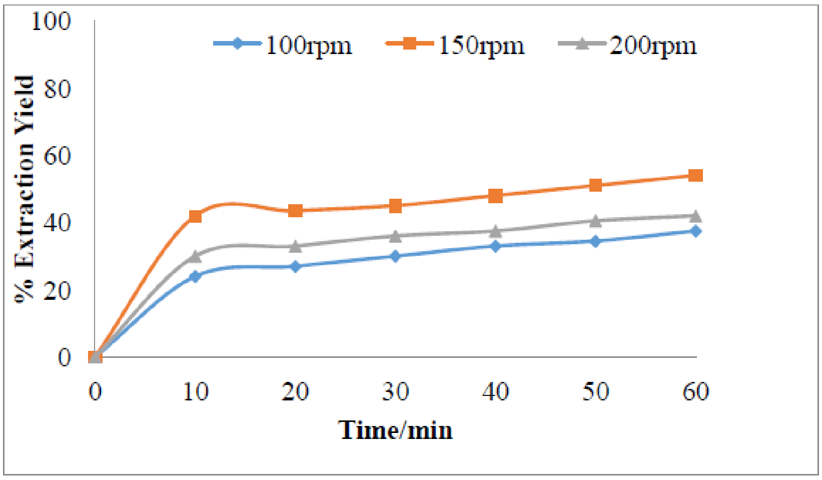

Figure 1: Effect of Agitation Speed on the Extraction Yield of Lawsonia Inermis at 50% methanol 50% water, SSR 1/15

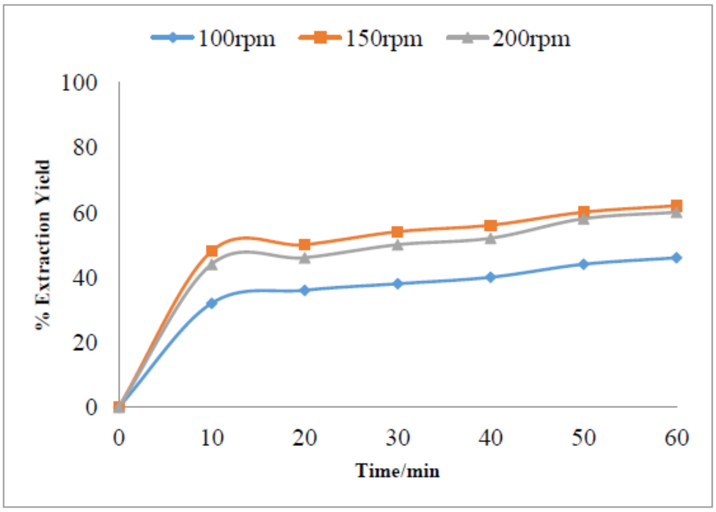

Figure 2. Effect of Agitation Speed on the Extraction Yield of Lawsonia Inermis at 50% methanol 50% water, SSR 1/20

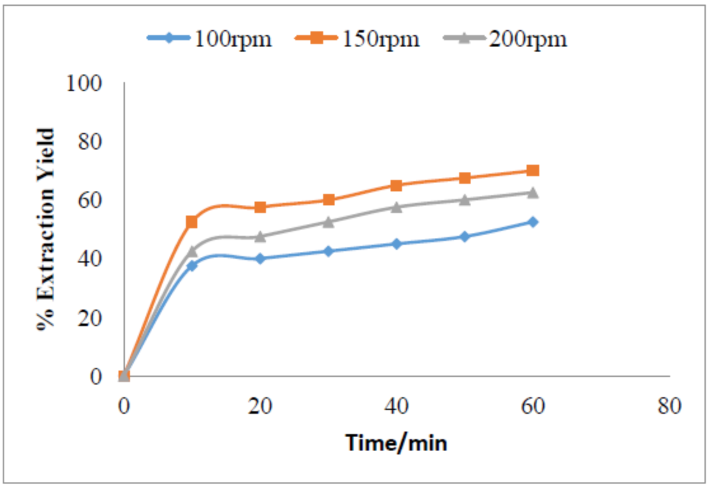

Figure 3. Effect of Agitation Speed on the Extraction of Lawsonia Inermis at 50% methanol50%water.SSR1/25

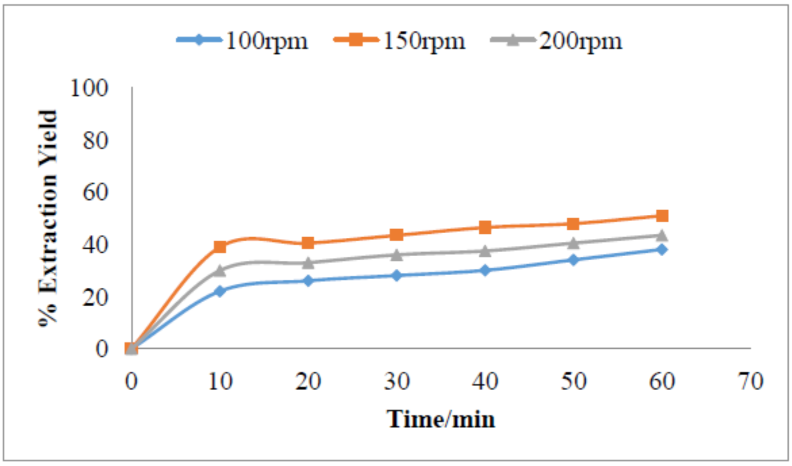

Fig 4. Effect of Agitation Speed on the Extraction of Parkia Biglobosa at 50% methanol 50% water, SSR 1/15

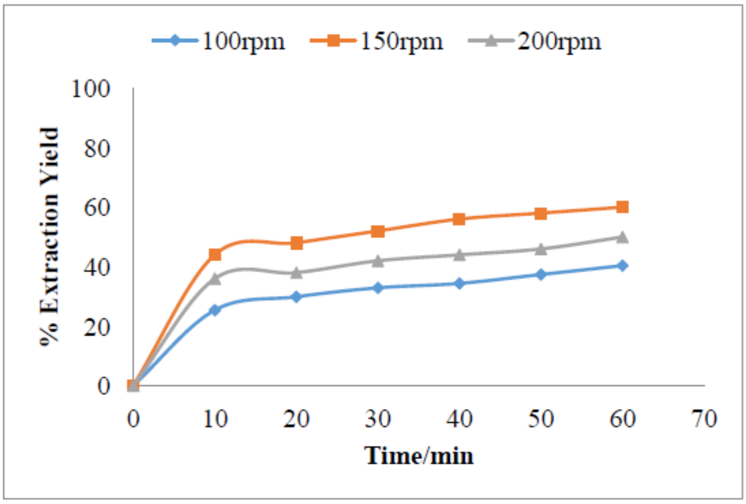

Figure 5. Effect of Agitation Speed on the Extraction Yield Parkia Biglobosa at 50% methanol 50% water, SSR 1/120

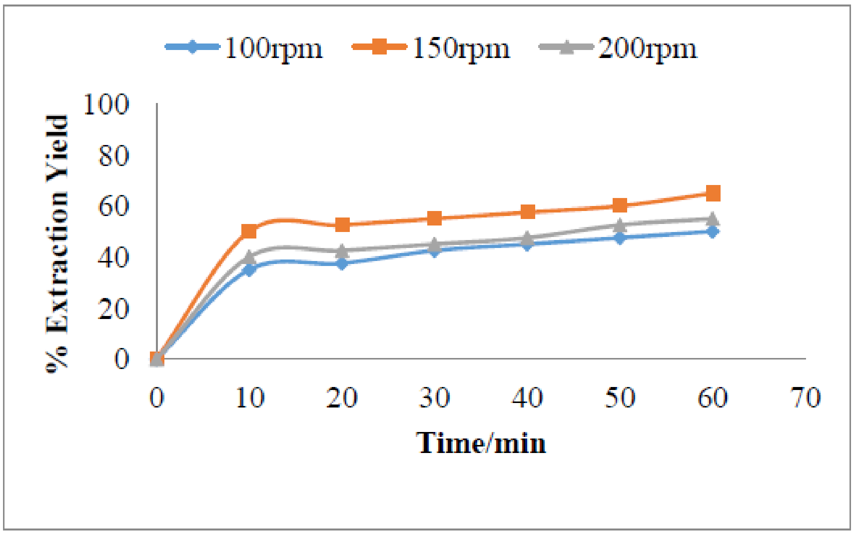

Figure 6. Effect of Agitation Speed on the Extraction Yield Parkia Biglobosa at 50% methanol 50% water, SSR 1/25.

When the effect of agitation speed was examined for Parkia Biglobosa, the results in Figs 4-6 were obtained. A high yield was obtained and close to what is reported for Lawsonia leaves at same speed and SSR. When cocholspermum tinctorium was considered (Figs 7-9), smaller amount of the extract was obtained at low speed (150rpm) , and SSR 1/25. However,

for all plant materials examined, higher speed (t 200rpm) did not produce more of the extract than the low speed . This could be as a result of insufficient time for solute –solvent interaction and high weight for rotating the magnetic stirrer.

In addition, the observed difference in the amount of extract in the three plants viz: Lawsonia inermis {70%}, Parkia Biglobosa {65%} and Cochlospermum {57%} could be attributed to the differences in the texture of cell wall of plant materials. While Lawsonia inermis is a leafy material, Parkia biglobosa is a tough husk and Cochlospermum tinctorium is a fibrous material that are difficult to break and extract the coloring material using similar solvent mixture.

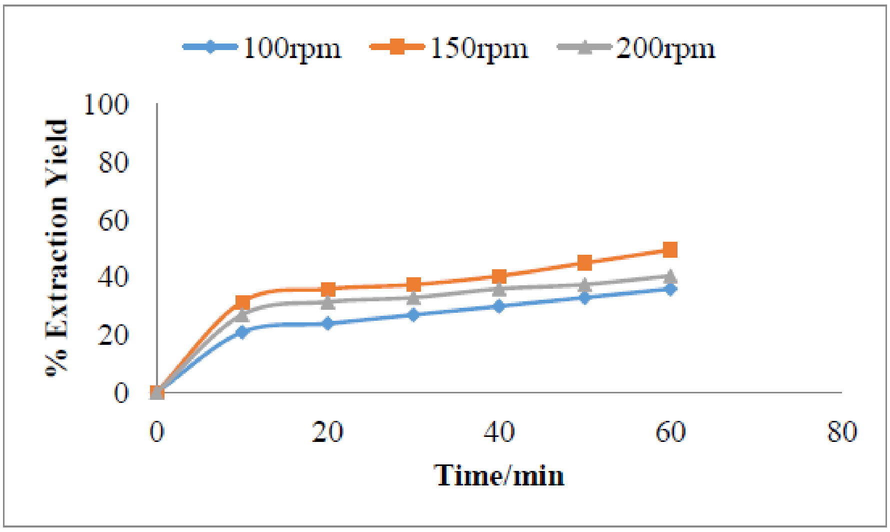

Figure 7. Effect of Agitation Speed on the Extraction Yield CochlospermumTinctorium at 50% methanol 50% water, SSR 1/15

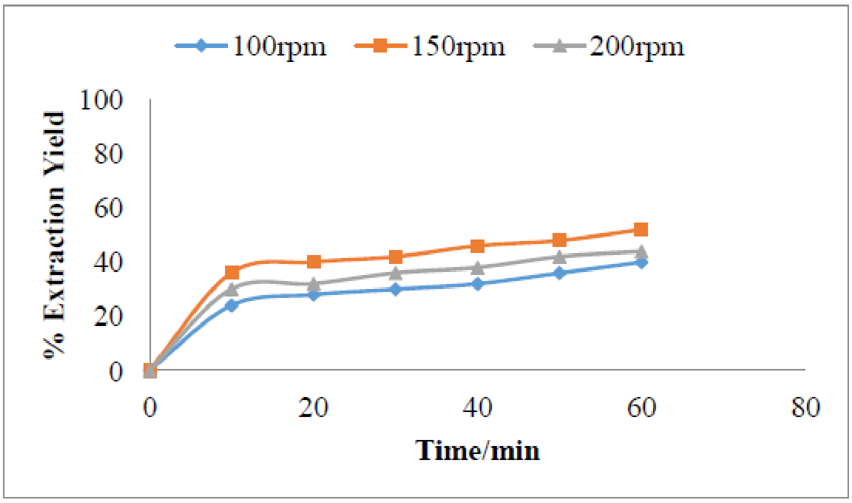

Figure 8. Effect of Agitation Speed on CochlospermumTinctorium Extraction yield during MS at 50% methanol 50% water, SSR 1/120

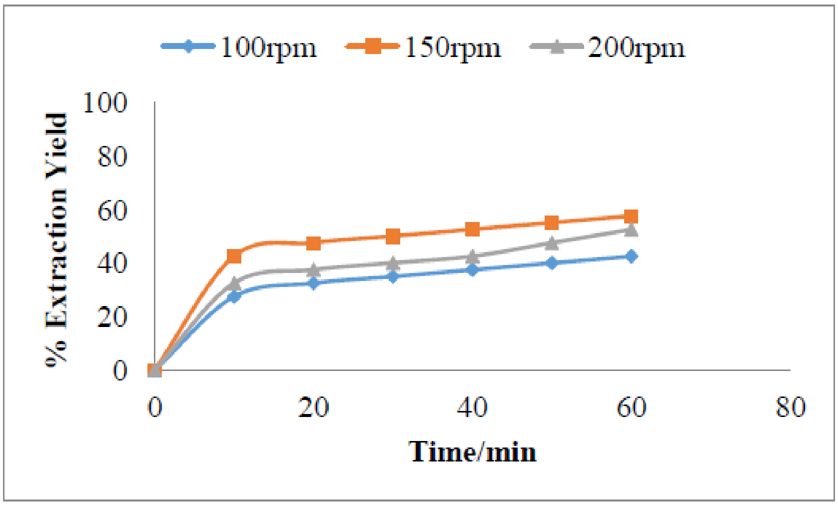

Figure 9 . Effect of Agitation Speed on the Extraction Yield of CochlospermumTinctorium at 50% methanol 50% water, SSR 1/25

Effect of Microwave Powers on the Extraction of Dye from Plant Matrices

The results of the effect of microwave power on the extraction efficiency of dye from Lawsonia inermis are given in Figs10-12. The highest yield recorded was 87% at high power and SSR 1/25 and a minimum of 13% at low power and SSR 1/15.

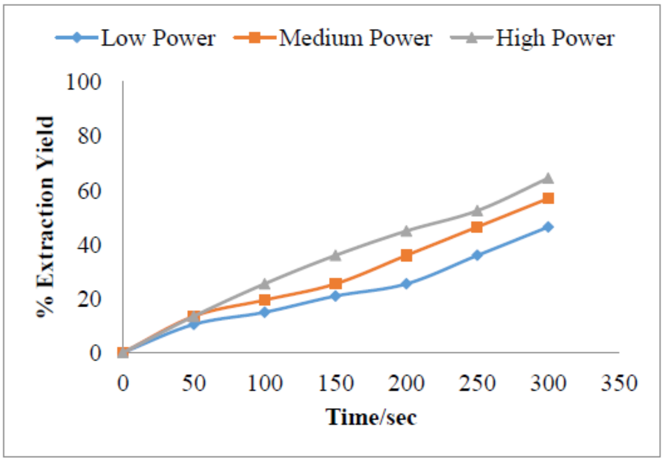

Figure 10. Effect of Microwave Power on the Extraction Yield of Lawsonia Inermis at 50% methanol 50% water, SSR 1/15

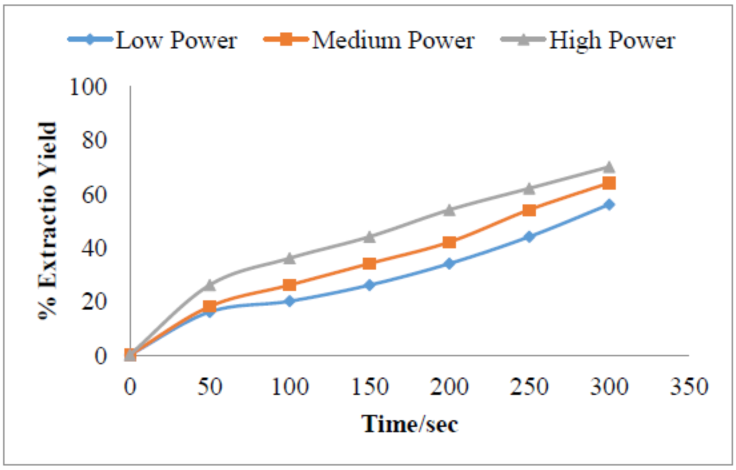

Figure 11. Effect of Microwave Power on the Extraction Yield of Lawsonia Inermis at 50% methanol 50% water, SSR 1/20

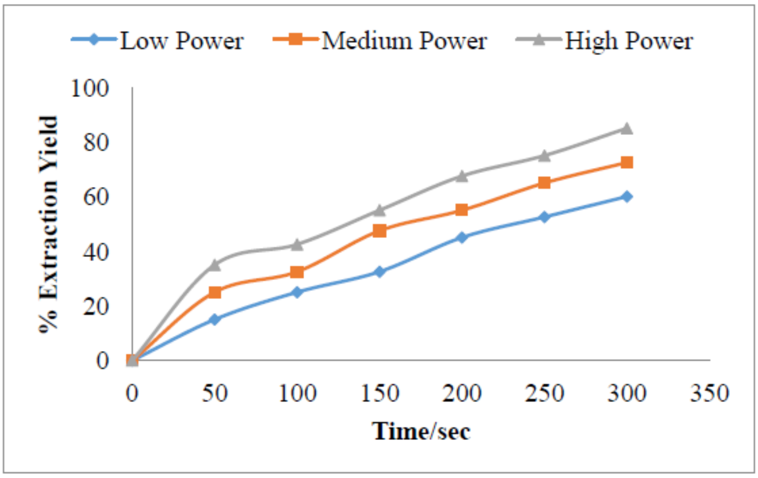

Figure 12. Effect of Microwave Power on the Extraction Yield of Lawsonia Inermis at 50% methanol 50% water, SSR 1/25

When the experiment was repeated with Parkia Biglobosa , percent extraction was as high as in Lawsonia inermis (Figs13-15). There are also similarities in performance between the two plant matrials at low and medium power and SSR.

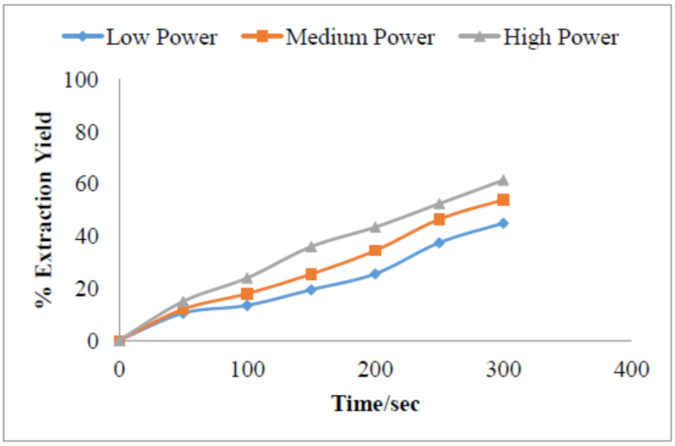

Figure 13. Effect of Microwave Power on the Extraction Yield of Parkia Biglobosa at 50% methanol 50% water, SSR 1/15

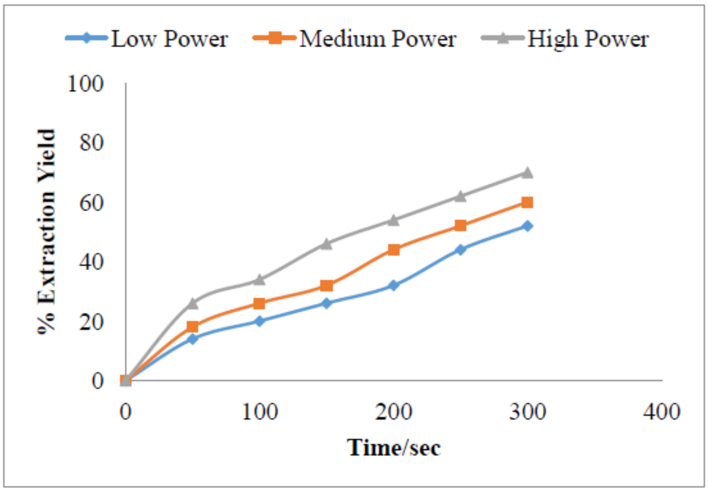

Figure 14. Effect of Microwave Power on the Extraction Yield of Parkia Biglobosa using 50% methanol 50% water, SSR 1/20

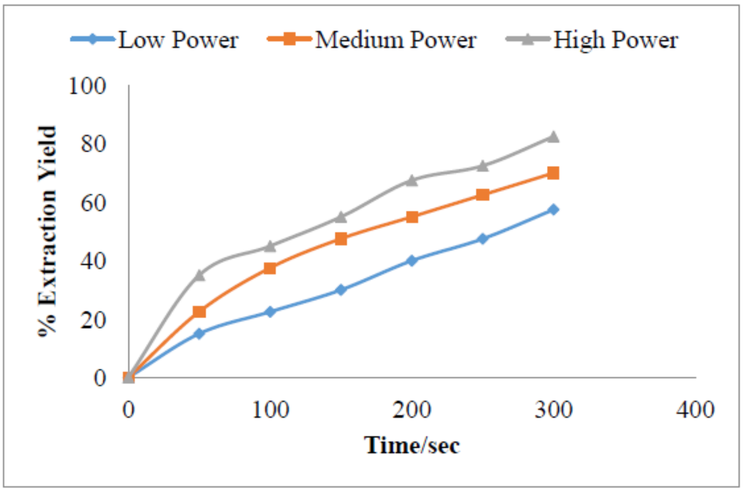

Figure 15. Effect of Microwave Power on the Extraction Yield of Parkia Biglobosa at 50% methanol 50% water, SSR 1/25

The short irradiation time is enough to rapture the cell wall at that temperature to extract the colorants. In all cases, elevated power results in the greatest extraction efficiency. The short time when compared to magnetic stirrer is to avoid at decomposition at high temperature. Note also, the maximum extraction was achieved within 5 minutes compared to 1 hour for Magnetic stirrer technique.

3.4 Effect of Solid to Solvent Ratio on the Extraction Yield of Colorants from Plant Matrices.

The effect of solid to solvent ratio 1/15, 1/20, 1/25 was investigated at different agitation speed and microwave power for all the plant materials (Figs 16-21) and the results showed that the amount of dye extracted for all the plants increase in intensity from pale to deep color as the SSR increases. The highest yield was achieved at SSR 1/25 at high power and agitation speed for all the plant matrices. low vlues exist when magnetic stirrer was employed.

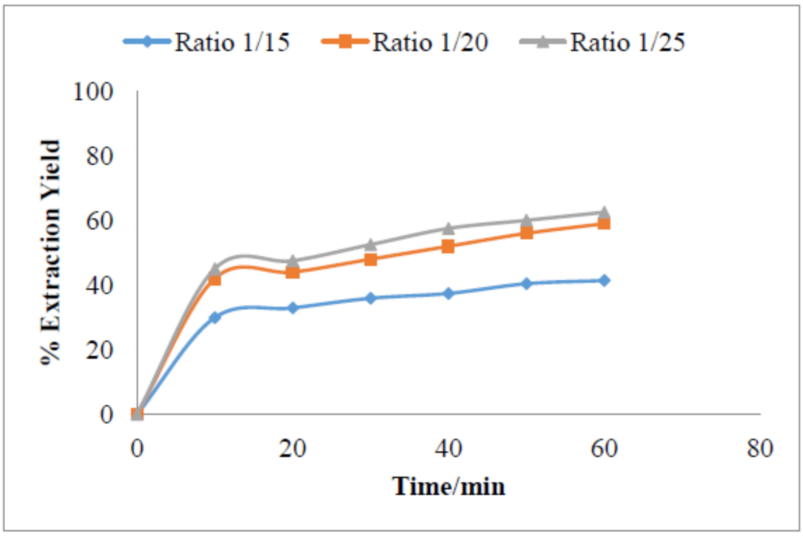

Figure16. Effect of Solid to Solvent Ratio on the Extraction Yield of Lawsonia Inermis at 50% Methanol, Agitation Speed 200rpm

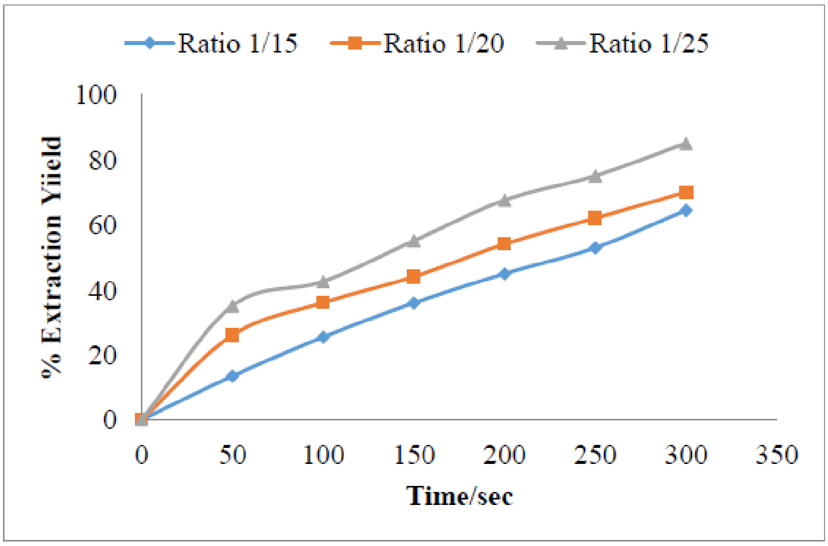

Figure 17. Effect of Solid to Solvent Ratio on the Extraction Yield of Lawsonia Inermis at 50% Methanol, High Microwave Power

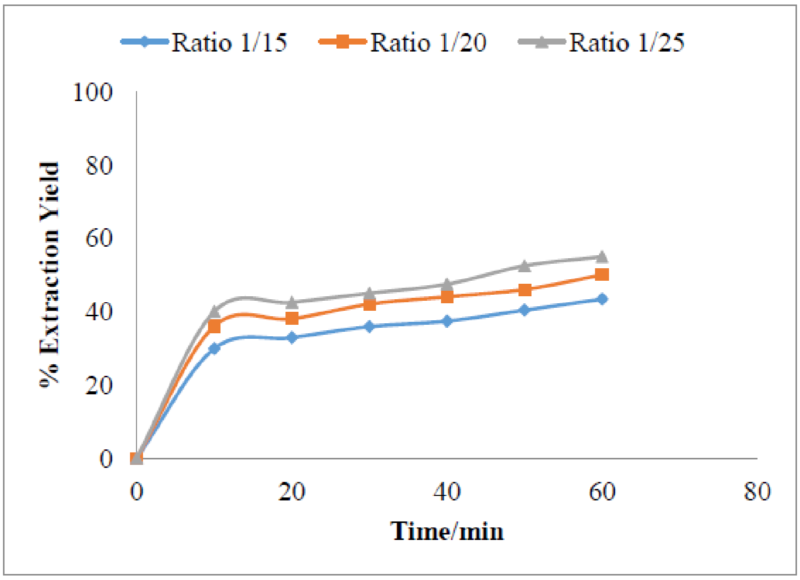

Figure 18. Effect of Solid to Solvent Ratio on the Extraction Yield of Parkia Biglobosa at 50% Methanol, Agitation Speed of 200rpm

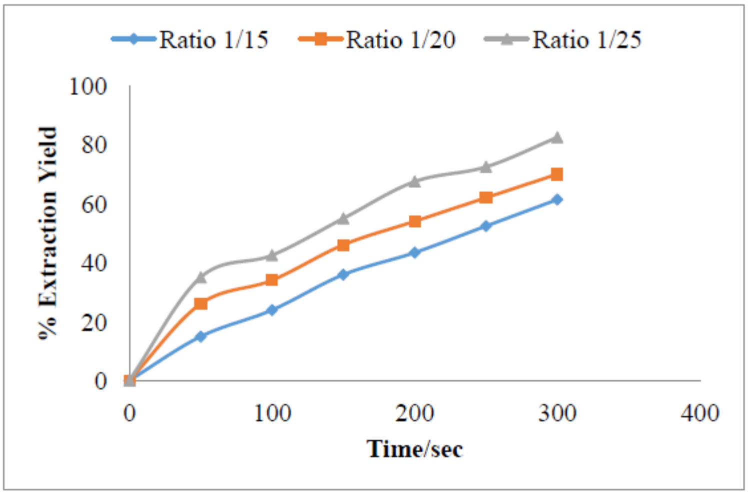

Figure 19. Effect of Solid to Solvent Ratio on the Extraction Yield of Parkia Biglobosa at 50% Methanol, High Microwave Power

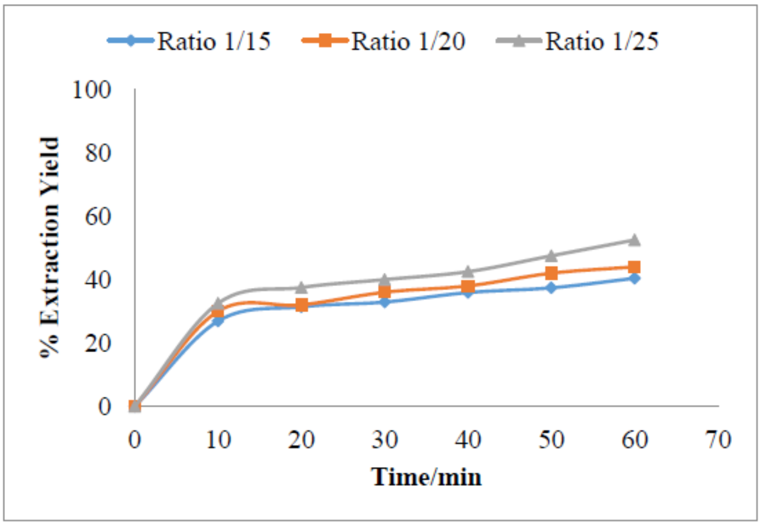

Figure 20. Effect of Solid to Solvent Ratio on the Extraction Yield of CochlospermumTinctorium at 50% Methanol, Agitation Speed of 200rpm

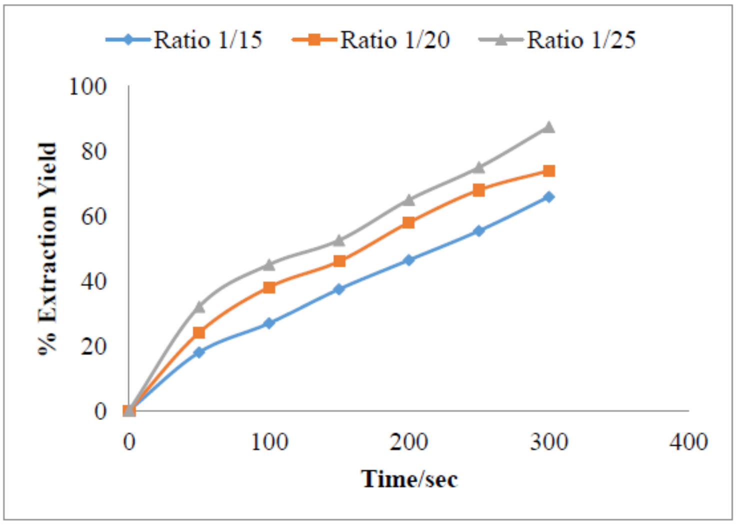

Figure21. Effect of Solid to Solvent Ratio on the Extraction Yield of Cochlospermum Tinctorium at 50% Methanol, High Microwave Power

The general trend seen in the three plant materials may be due to low dissolution of substrate at low solvent volume interaction. However as the SSR increases, the amount of substrate rise and the surface area of contact increase and therefore release more colorants.

Effect of Time on the Extraction of Dyes
When the effect of time on the extraction of dye from all plants was considered , the results showed increase in percent extraction with time in all cases. However, the maximum extraction was achieved within 5 minutes with microwave while magnetic stirrer took 1 hour to achieve similar performance. This allows sufficient time for swelling of the plant cell wall and solvent to reach the dye. The increase in power as a result of elevation of temperature causes the dissolution of dye and rapture of cell wall within short time compared to magnetic stirrer. Elongation of time greater than 5 minutes could lead to denaturation and evaporation of solvent to ignite fire which is hazardous .

Effect of Solvent Composition on the Extraction Yields of Dyes from Plant Matrices Where the effect of solvent composition on the extraction yield of dye from Lawsonia inermis, Parkia Biglobosa and Cochlospermum tinctorium was investigated , the maximum extraction was at 50:50% (v/v) methanol/water mixture for all species. The trend was similar for for the microwave method at different SSR ,low, medium and high powers. However when solubility was considered, the order was Lawsonia inermis< Parkia Biglobosa < Cochlospermum tinctorium. This is as a result of the influence of power increase and solvent polar efficiency. It is also observed that increase in agitation speed or microwave power for solvent composition 30% : 70%, 70% :30% (methanol : water) mixture is less effective than the 50:50 (v/v). At higher methanol composition the extraction falls because the dissolution of dye has reached equilibrium value and cannot therefore dissolve more above the optimum value of 50:50% (v/v).

IV. Conclusion

The results of this work shows that microwave power is a very good technique in the extraction of dye from different plant materials as it reduces the time required for the extraction compared to other classical techniques. Different solvent mixture, agitation speed on magnetic stirrer, solid to solvent ratio and different powers of microwave were used using particle size of 0.45 to determine the highest extraction yield of Lawsonia inermis, Parkia Biglobosa and Cochlospermum which were obtained at 50% methanol: 50 % water, 150rpm, 1/25 SSR and high microwave power.

Compliance with ethics Requirements
This paper does not contain studies with animal subjects

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Declaration of interest
No conflict of interest

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