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Removal of Lead from polluted water by using bio-sorbents extracted from ClitoriaTernatea and applying thermodynamics and kinetic techniques.

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Abstract

Lead metal is used widely because of its mechanical properties. Lead is poisonous to human beings. Clitoria ternatea plant has lot of medicinal values. The plant leaves, stems, fruits and flowers were washed with distilled water and dried in sun light. The dried materials of the plant are reduce the size of the particles below75µ and activated at 100^0C using oven. At pH 6, at the period of 7 days with maximum adsorbent dose of dry leaves powder has 85.01% of adsorption of Lead. Two different adsorption isotherms such as Freundlisch and Langmuir are analyzed and correlation coefficient and dimensionless separation factor (R_L) values, Clitoria Ternatea dry leaves powder has (R^2 =0.994)**.** Pseudo-first-order and pseudo-second-order equations are applied to identify the rate and kinetics of adsorption process. The adsorption process has good correlation coefficient of Clitoria Ternatea dry fruit is R^2 values is 0.996 with pseudo-first order model. Removal of Lead from industrial water shows maximum adsorption of 75.67% by dry leaves powder. The biosorbent powders are structurally analyzed with FT-IR spectroscopy.

Key words: Lead, bio sorbent, adsorption, Clitoria ternatea, isotherms, kinetic studies and FT-IR.

1. Introduction

Lead is a heavy metal with symbol Pb and atomic number 82. It is soft, malleable and ductile. It is a bluish-white[1] colour metal when it is exposed to air turns to dull gray. It is corrosion resistant[2] and when melted into a liquid has a shiny chrome-silver look. Lead metal is used widely because of its mechanical properties like high density, low melting point, ductility, inertness and inexpensive[3].

Nowadays, over half of the lead produced is used as electrodes in lead-acid car batteries. Its high density and resistance to corrosion makes it useful as the ballast keel of sail boats[4] and as scuba diving weight belts[5]. Lead is also used in the construction industry for roofing, cladding, gutters and glazing bars for stained glass[6,7]. Lead is still widely used to make statues and sculptures[8]. It is used to make bullets and is also used in radiation shields around X-ray equipment[9]. Lead has been used as a paint additive, in face whitening make-up, in water pipes, preservative for food and drinks, pesticide, and in paint used on children's toys.

Lead is poisonous to human beings if inhaled or swallowed. Lead poisoning can have a major effect on the body's brain, kidneys and nervous system[10]. It can damage the body's organs and can cause weakness in the body's joints. Some symptoms of lead poisoning include nausea, vomiting, extreme tiredness, high blood pressure, and convulsions (spasms). Over a long period of time, children often suffer brain damage. They lose the ability to carry out normal mental functions. Lead poisoning occurs due to contamination of soil and water nearby industries, usage of lead pipes, lead paint and residual emissions from leaded gasoline[11].

It is very essential to remove Lead from the polluted water because of above adverse effects, to prevent environmental pollution and human beings. Few research articles are available for the removal of Lead from the polluted water. Wolvetron B.C and Mc donald R.C. at al[12] have investigated removal of lead and mercury by water hyacinths (Eichhornia crassipes) (Mart.) Solms and alligator weeds (Alternanthera philoxeroides). Uptake of arsenic, cadmium, lead and mercury from polluted waters by the water hyacinth Eichornia crassipes by Francis E. Chigbo at al[13], biosorption: An eco-friendly alternative for heavy metal removal by HK Alluri at al[14] and Azolla pinnata r.br. and lemna minor l. for removal of lead and zinc from polluted water. so it is essential to develop simple and new methods with low cost by using bio-sorbents like weeds to prevent the environment and living organisms from poisoning of Lead by using simple analytical volumetric analysis. There are various techniques available to remove heavy metals like ion exchange, membrane filtration, electrolysis and coagulation[15] but they are high cost, sludge generation and selectivity of metals. Bio sorption technique is an eco-friendly, sustainable, rapid, easily available and low cost.

1.1 Plant description.

Clitoria ternatea (Sankupushpam or Butterfly pea) is a perennial climber found in tropical regions of Asian Continent and is believed to be native of Asia. The plants are seen much adaptive to various ranges of temperatures and humidity. They are tolerant to frost and dry conditions. The plant is a perennial herb seen all round unnoticed in between the shrubs and small trees, most of the time the flowers are seen peeping from the greenery of the plant. The flowers are lovely and are snowy white, dark blue, luminescent blue, violet with single or double petals. Since no part of this plant is poisonous, all parts are used either in cooking or in medicines.

Figure-1. Clitoria Ternatea (Sanku pushpam) plant

Coming to medicinal uses it is to be noted that the plant and its parts are used to prepare a variety of medicinal extracts in Ayurveda. The medicines are used both internally as well as externally. The plant is generally found to be having antifungal properties. Plant extracts are known to be alleviators of pain and swellings and for treating circulatory system as it has blood purifying and haemostatic properties and also for treating smallpox.

Hence Clitoria Ternatea plant was selected for this experiment due to its easy availability and having lot of medicinal values.

1.2 Objective of this method

- Stem, fruits, leaves and flowers of the Clitoria Ternatea plant in dry and ash powders were used as bio-sorbents to remove Lead from the polluted water.
- To calculate the pH verses percentage of removal of Lead.
- To calculate the time verses percentage of removal of Lead.
- To calculate the adsorbent doses verses percentage of removal of Lead.
- The effect of temperature verses percentage of removal of Lead
- To study the mechanism by adsorption isotherms and kinetic models
- To determine the structural analysis of bio-sorbents before and after adsorption by using FT-IR spectroscopy.

2. Adsorption experiment

Apparatus

Analytical balance, reagent bottles, volumetric flasks, conical flasks, pipettes, burettes, measuring jars, burette stand and Hot air oven.

Chemicals

Lead nitrate, potassium chromate, acetic acid, sodium acetate, sodium thiosulphate, Hydro chloric acid(HCl) and starch powder.

2.1 Preparation of biosorbents powder

The leaves, stems, fruits and flowers cut from Clitoria Ternatea plant were washed with distilled water and dried in sun light. The dried materials were crushed and meshed to reduce the size of the particles below75µ and activated at 100^0C using oven. Ash adsorbents were prepared by burning the plant materials as discussed above.

2.2 Preparation of lead sample solution

1.0 gram of Lead nitrate is dissolved 1000mL of distilled water to make the concentration of 1000 ppm.

Preparation of Potassium chromate solution

1.0 gram of Potassium chromate is dissolved in 1000 mL of water to get the concentration of 100 ppm.

Preparation of acidic buffer:

50mL of Acidic acid is dissolved 70mL of water contains 4 grams of sodium acetate.

Preparation of hypo solution:

15 grams of Sodium thio sulphate (hypo) is dissolved in 1000mL of distilled water.

Preparation of starch solution:

1.0 gram of starch powder is dissolved in 2 to 3mL distilled water and poured this content into 100 mL boiling water. Stirred the contents with glass rod and made into a uniform solution and cool to room temperature.

Preparation of stock solution

500 mL reagent bottles were thoroughly washed and rinsed with distilled water and each bottle is filled with 250 mL of 1000 ppm Lead nitrate solution. In reagent bottles different doses of bio-sorbents (dry and ash powders) are added and shaken well with frequent times and kept it for better absorption of Lead. The contents are thoroughly shaken and filtered through Wattmann filter paper through funnel into a cleaned reagent bottle. The filtrate is

stored in cold and dry place for further experiment. The powders of bio-sorbents before and after absorption were examined with FT-IR spectrophotometer to determine the change of the spectra due to absorption of Lead.

Procedure

From bio sorbent solution, 20mL is pipetted out into a clean conical flask. To this same quantity of potassium chromate solution is added yellow color precipitate is formed. The precipitate is dissolved with concentrated HCl and 2mL of acidic buffer is added. To this one gram of potassium iodide is added and closed the conical flask and kept in dark place for 5 minutes. After 5 minutes the contents are titrated against hypo until pale yellow color is reached. To this 1mLof starch indicator is added and titrated against hypo solution till the contents in the flask turns to pale green color. The end point is noted from the burette reading. The same procedure is repeated with blank and as well as with stock solutions of various bio sorbents.

3. Results and discussions

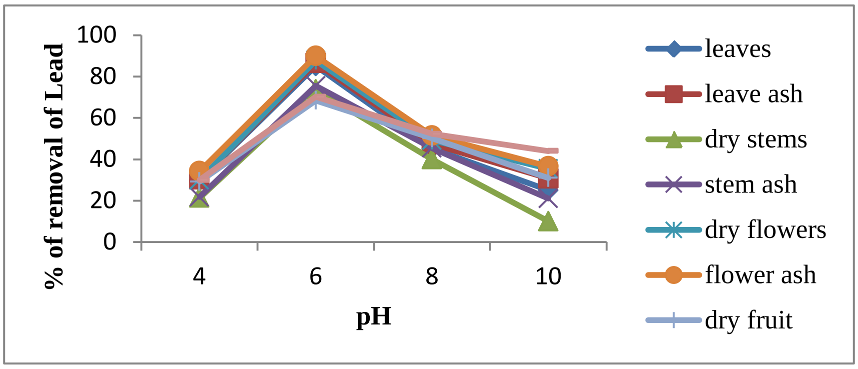
The removal of Lead from polluted water was investigated by changing the various physicochemical parameters like pH, time, adsorbent doses and temperature.

3.1 Effect of pH on adsorption of Lead

The adsorption of Lead is observed maximum at pH 6 range. Leaves, leaves ash, stems and fruits powders were shown maximum adsorption. The adsorption falls gradually by increasing the pH from 6 to 10. At pH 6, leaves have highest 85.01% of adsorption where as leaves ash and stems have 78.94% of adsorption and fruits have 77.19% adsorption of Lead. At pH 10, leaves ash has shown least adsorption of lead that is 13.25%. The percentage of removal of Lead by bio sorbent powders is presented in theTable-1. The adsorption isotherm is shown in the Graph-1.

Table-1 Effect of pH on % of removal of Lead

pH	dry leaves	leave ash	dry stems	stem ash	Dry flowers	flower ash	dry fruit	fruit ash
4	26.07	29.57	21.01	21.78	30.35	24.90	21.78	29.57
6	85.01	78.94	78.94	70.17	73.68	73.68	77.19	73.68
8	50.45	49.91	51.56	48.41	49.26	48.41	50.28	50.10
10	14.53	13.25	18.84	15.42	15.35	15.89	16.02	15.29



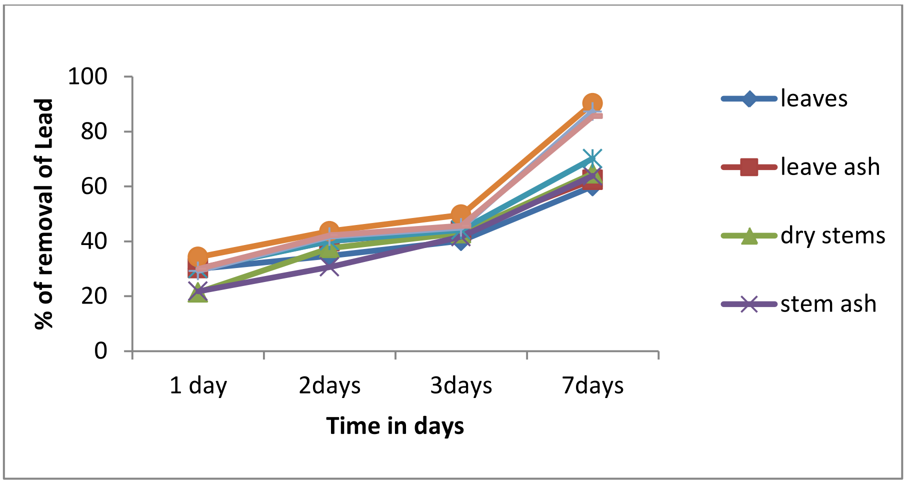
Graph-1 Effect of pH on % of removal of Lead

3.2 Effect of time on adsorption of Lead

By increasing the time from 1 to 7 days by frequent shaking dry leaves has maximum adsorption of 85.01% followed by leaves ash and dry stems have 78.94% of adsorption of Lead after 7 days. It has been observed that by increasing the time with different bio-sorbents, the removal of Lead has increased gradually. The % of removal of lead by bio sorbent powders is shown in theTable-2. The absorption isotherm is shown in the Graph-2.

Table-2 Effect of Time on % of removal of Lead

Time in days	dry leaves	leaves ash	dry stems	stems ash	dry flowers	flowers ash	dry fruits	fruits ash
1	24.39	23.26	25.01	23.88	26.92	25.71	24.63	23.87
2	35.18	34.86	36.08	33.17	36.24	35.91	34.96	33.25
3	42.26	41.17	43.27	42.69	44.79	41.52	41.47	40.58
7	85.01	78.94	78.94	70.17	73.68	73.68	77.19	73.68



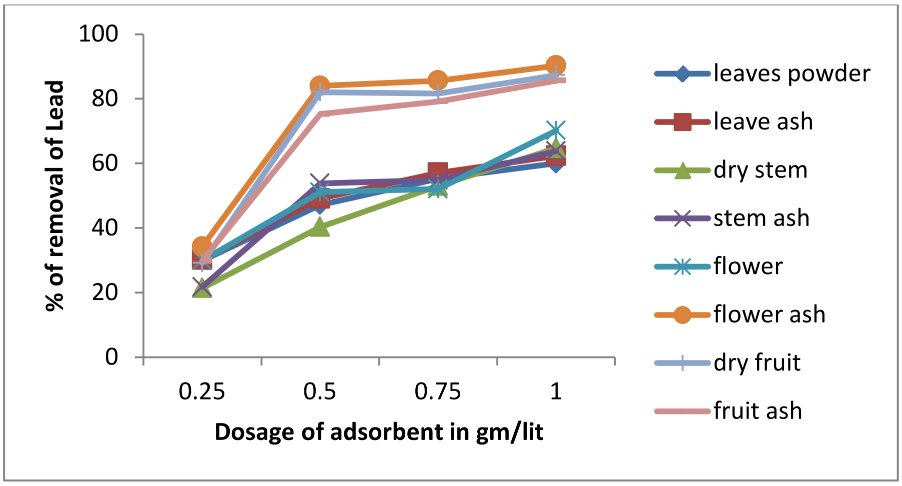
Graph-2 Effect of time on % of removal of Lead

3.3. Effect of adsorbent doses on adsorption of Lead

Initially 0.25gms of bio-sorbents are used for the elimination of Lead. Fruits have 28.82% of absorption. By increasing the doses from 0.25gm to 1gm, the elimination of Lead has also increased gradually, leaves has maximum adsorption of 85.01% followed by leaves and stems have 78.94% of adsorption. The % of removal of Lead by bio sorbent powders is presented in theTable-3. The adsorption isotherm is shown in the graph-3.

Table-3 Effect of absorbent doses on % of removal of Lead

Absorbent doses	dry leaves	leaves ash	dry stems	stems ash	dry flowers	flowers ash	dry fruits	Fruits ash
0.25	21.39	20.08	22.32	20.61	28.77	24.98	28.82	27.56
0.5	45.11	44.72	43.59	42.27	51.06	44.18	40.03	39.93
0.75	72.45	67.25	65.84	64.25	69.90	68.72	71.56	69.45
1	85.01	78.94	78.94	70.17	73.68	73.68	77.19	73.68



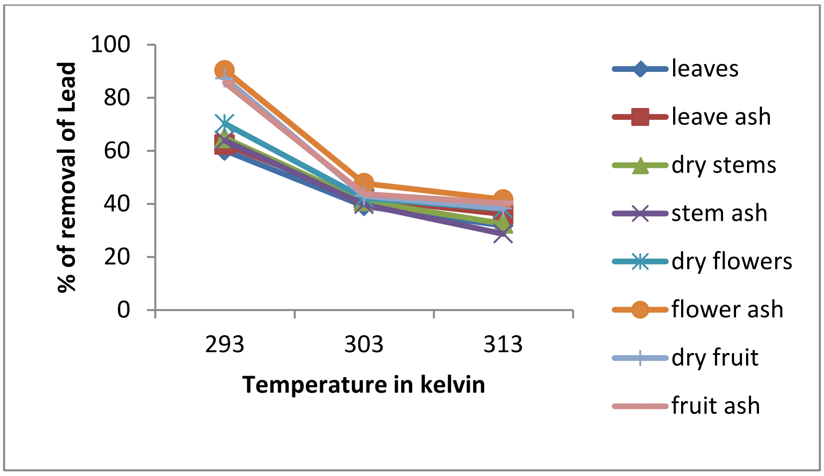
Graph-3 Effect of adsorbent doses on % of removal of Lead

3.4 Effect of temperature on the adsorption of Lead

Leaves shown maximum 85.01% of absorption at temperature of 293K followed by leaves ash and stems at 78.94% of absorption. By increasing the temperature of bio-sorbents solution by heating, it is observed that adsorption rate is decreased with increase in the temperature at 313K. The % of removal of Lead by bio sorbent powders is presented in theTable-4. The absorption isotherm is shown in the Graph-4.

Table-4 Effect of temperature on % of removal of Lead

Temperature	dry leaves	leaves ash	dry stems	stems ash	dry flowers	flowers ash	dry fruits	fruits ash
293K	85.01	78.94	78.94	70.17	73.68	73.68	77.19	73.68
303K	40.12	39.0	41.17	38.72	42.9	41.27	42.0	40.73
313K	29.8	27.05	30.99	29.25	32.17	31.73	30.3	29.1



Graph-4 Effect of temperature on % of removal of Lead

4. Adsorption Isotherms

Freundlisch[16,] Langmuir[17]isotherms were used to evaluate the relation between the Lead concentrations remaining in the bulk solution to the amount of lead adsorbed at the solution interface at a constant temperature. Linear form of Freundlisch equation is log (q*e*) = log k*F* + (1/n) log C*e* and Linear form of Langmuir equation is C*e*/q*e*) = (a*L*/k*L*) Ce + 1/k*L*. According to Hall et al[18], the nature of the adsorption process is unfavorable (R*L* > 1), linear (R*L* = 1), favorable (0 < R*L* < 1) and irreversible (R*L* = 0) and the significant feature of the Langmuir isotherm model can be defined by the dimensionless separation factor, R*L* = 1/(1+ a*L*C*i*). The linear plots of these two adsorption isotherms are shown in Graph-5a to 5h and Graph-6a to 6h and isothermal constants along with the correlation coefficient values are presented in Table 5 and 6.

As the correlation coefficients (R^2 -values) is close to unity, it indicates the applicability of these two adsorption isotherms confirm the heterogeneous surface of the adsorbent and the monolayer coverage of Lead ion on the active carbon surface. The high correlation coefficient (*R*2 = 0.996) Clitoria Ternatea dry fruits values are favor to

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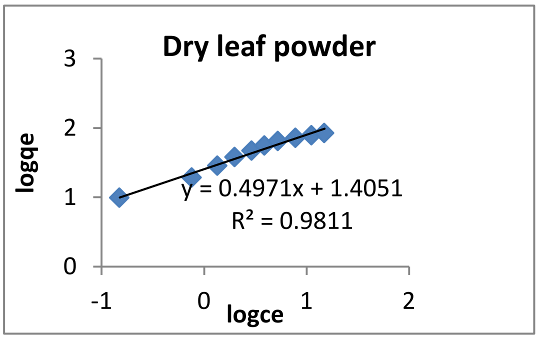
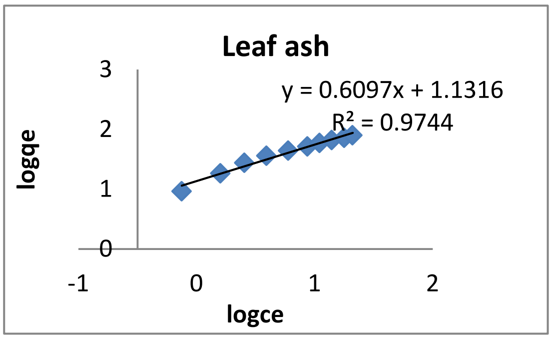
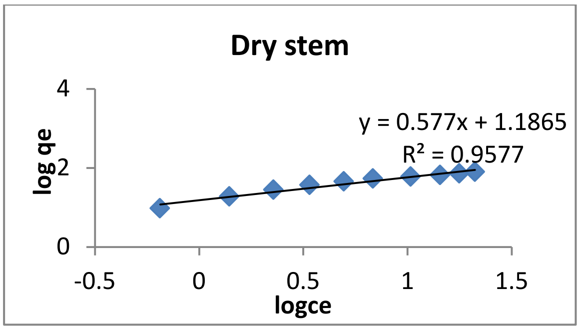
Freundlisch isotherm where as dry stem values are favor to Langmuir isotherm.



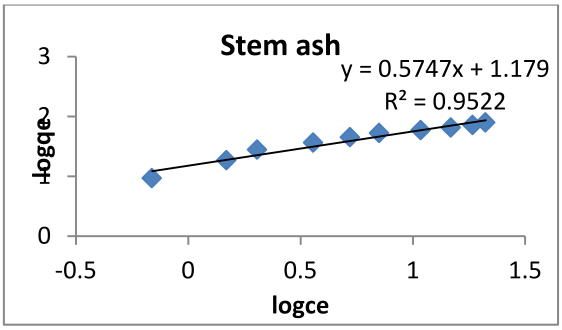
Graph -5a



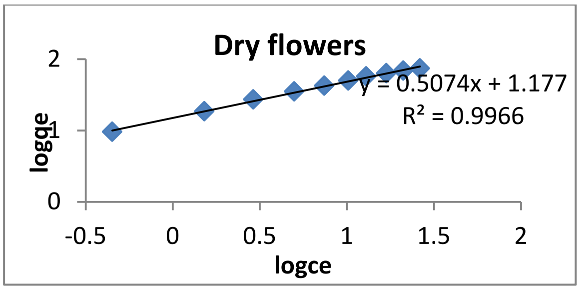
Graph-5b



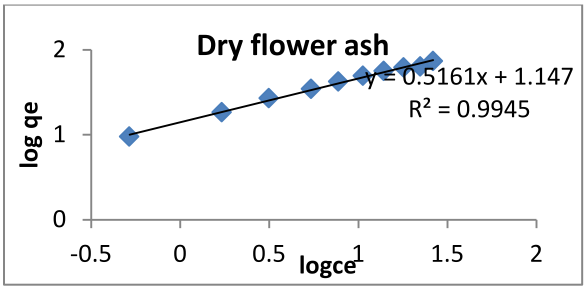
Graph-5c



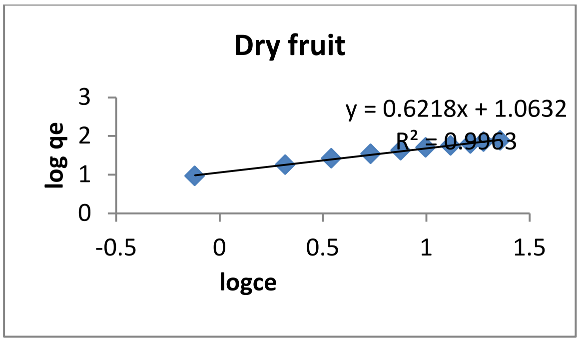
Graph-5d



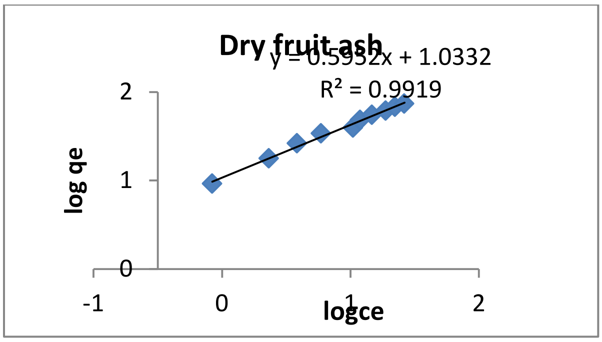
Graph-5e



Graph-5f



Graph-5g

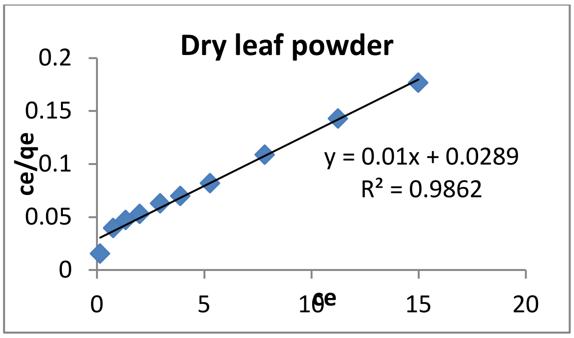


Graph-5h

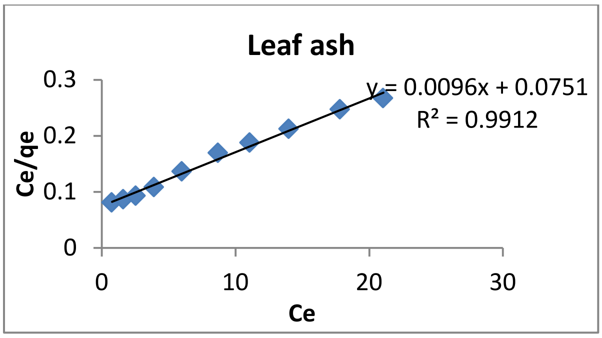
Graphs 5a-5h. Freundlisch adsorption isotherms of Clitoria Ternatea adsorbents

Table -5 Freundlisch adsorption isotherm parameters of Clitoria Ternatea

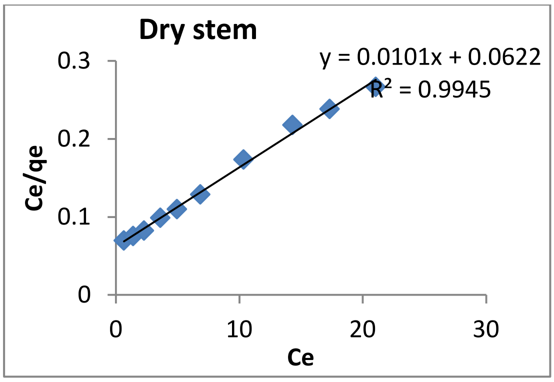
Sl.No	Name of the adsorbent	Slope	Intercept	R^2
1	Dry leaf powder	0.497	1.405	0.981
2	Leaf ash	0.609	1.131	0.974
3	Dry stem	0.577	1.186	0.957
4	Stem ash	0.574	1.179	0.952
5	Dry flowers	0.507	1.177	0.996
6	Dry flowers ash	0.516	1.147	0994
7	Dry fruit	0.621	1.063	0.996
8	Dry fruit ash	0.595	1.033	0.991



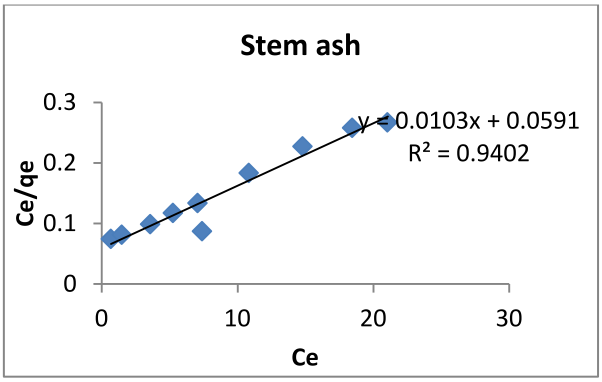
Graph-6a



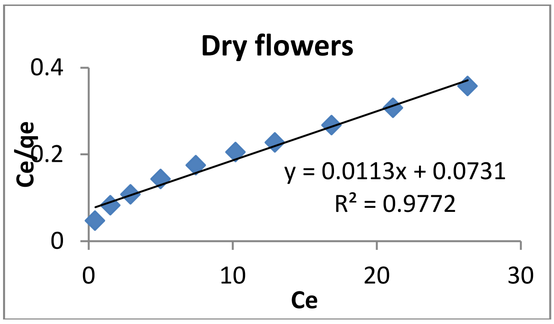
Graph-6b

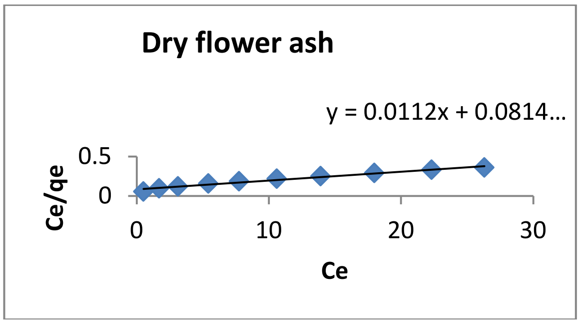


Graph-6c



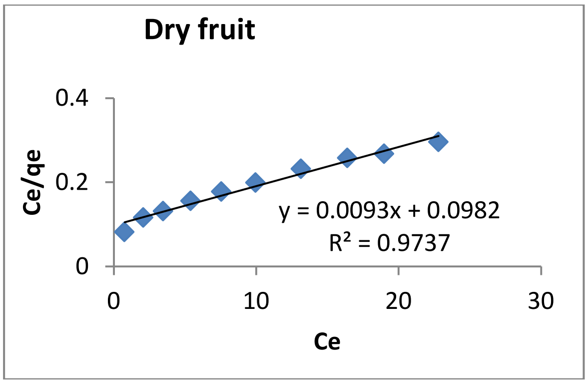
Graph-6d

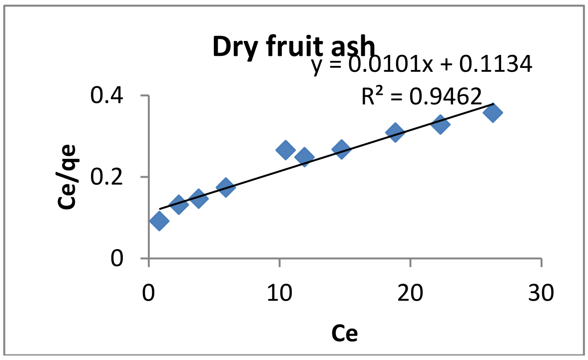




Graph -6e

Graph-6f





Graph-6g

Graph-6h

Graphs 6a-6h. Langmuir adsorption isotherms of Clitoria Ternatea adsorbents

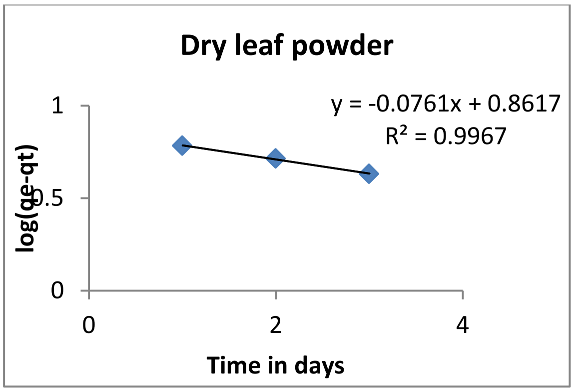
`Table – 6 Langmuir adsorption isothermal parameters

Sl. No.	Name of the adsorbent	R_L	Slope	Intercept	R^2
1	Dry leaf powder	0.0272	0.01	0.028	0.986
2	Leaf ash	0.0909	0.009	0.075	0.991
3	Dry stem	0.0584	0.01	0.062	0.994
4	Stem ash	0.05571	0.01	0.059	0.940
5	Dry flowers	0.06226	0.011	0.073	0.977
6	Dry flowers ash	0.06418	0.011	0.081	0.975
7	Dry fruit	0.09183	0.009	0.098	0.973
8	Dry fruit ash	0.1015	0.01	0.113	0.946

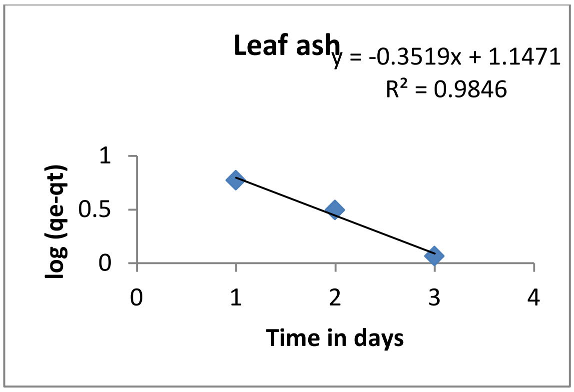
5 . Adsorption Kinetics

The rate and kinetics of adsorption of Lead ion 'onto' the Lead was studied with pseudo first-order model[19] and pseudo second-order model[20]. The pseudo first-order equation is $\log (q_e - q_t) = \log q_e - k_1 t/2.303$ and the pseudo second-order equation is $t/q_t = 1/k_2 - (1/q_e) t$; The linear plots of all these two kinetic models were as shown in Graph 7a to 7h and 8a to 8h respectively and rate constants along with the correlation coefficient values are presented in Table 7 and 8 respectively.

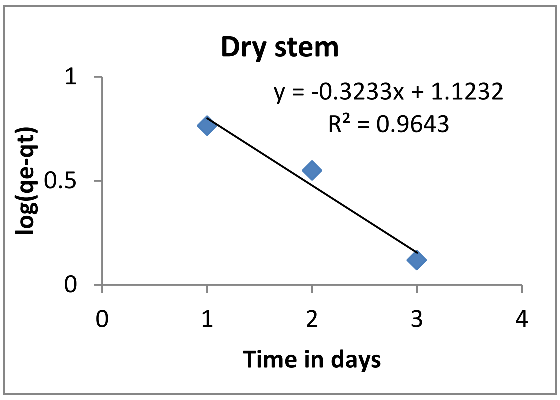
The applicability of the kinetic equations is compared from the correlation coefficient (R^2). From these two kinetic models, the correlation coefficient value for the pseudo first-order model is greater than pseudo second order kinetic model for most of the adsorbents of Clitoria Ternatea.



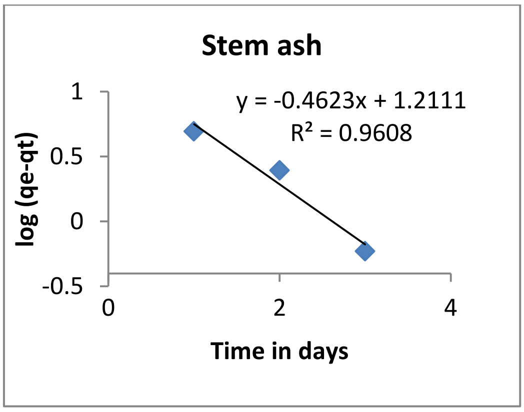
Graph-7a



Graph-7b



Graph-7c



Graph-7d

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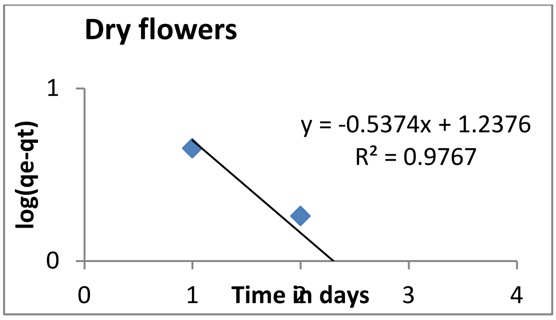

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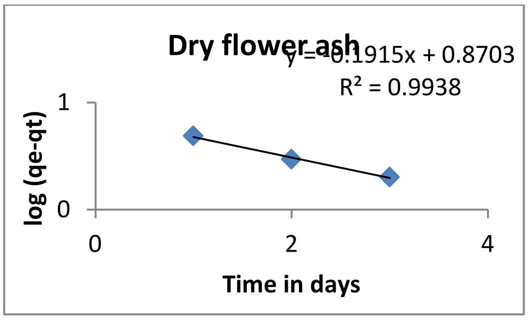
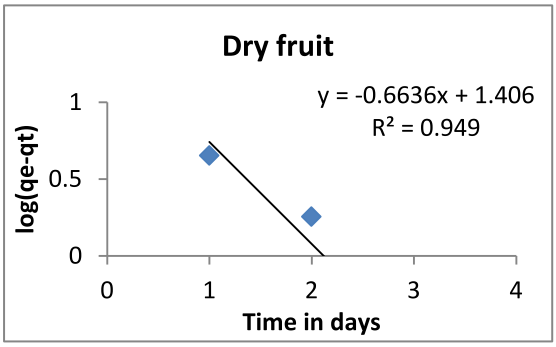
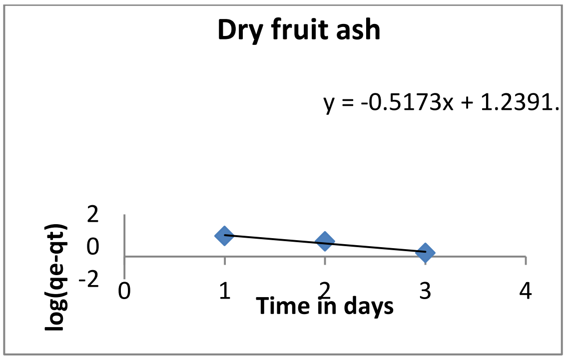


Graph-7f



Graph-7g



Graph-7h

Graphs 7a-7h: Pseudo first order for various adsorbents of Clitoria Ternatea

Table -7 Adsorption Kinetics-Pseudo first order reaction

Sl.No	Name of the adsorbent	Slope	Intercept	R^2
1	Dry leaf powder	-0.076	0.861	0.996
2	Leaf ash	-0.351	1.147	0.984
3	Dry stem	-0.	1.211	0.964
4	Stem ash	-0.462	1.211	0.960
5	Dry flowers	-0.537	1.237	0.976
6	Dry flowers ash	-0.191	0.870	0.993
7	Dry fruit	-0.663	1.406	0.949
8	Dry fruit ash	-0.517	1.239	0.960

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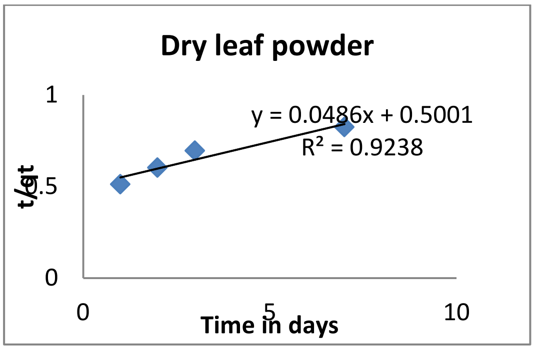
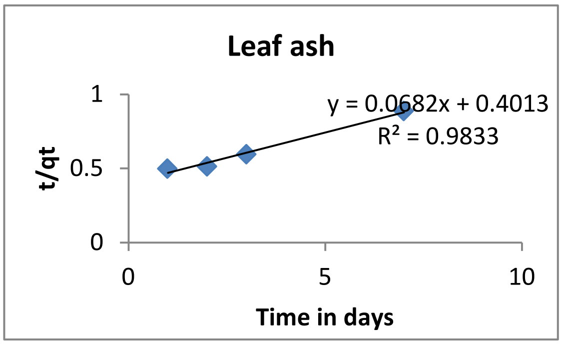
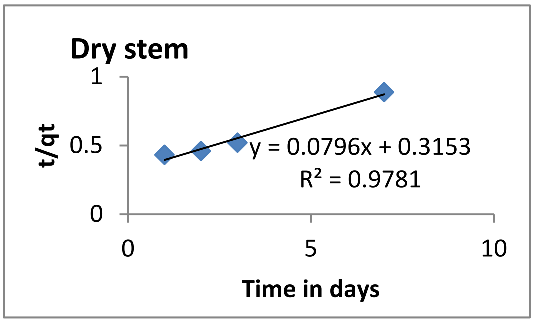


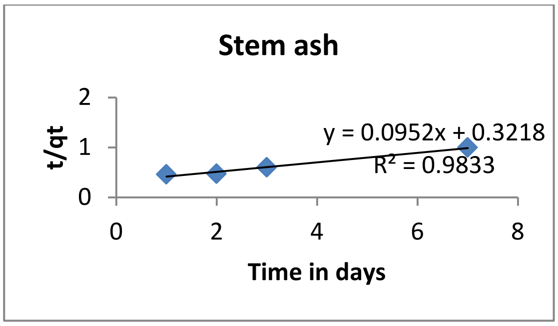
Graph-8a



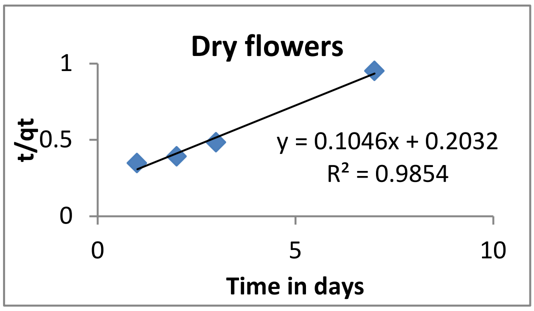
Graph-8b



Graph-8c



Graph-8d



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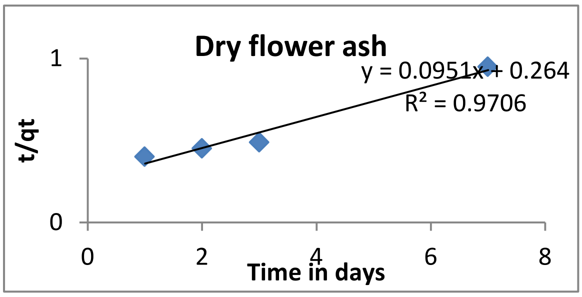
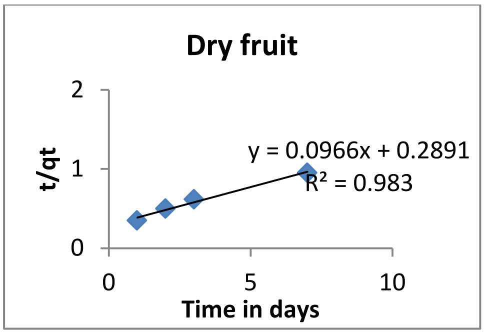
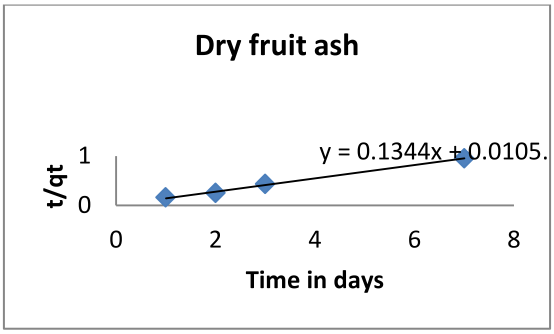


Graph-8f



Graph-8g



Graph-8h

Graphs 8a-8h: Pseudo second order for various adsorbents of Clitoria Ternatea

Table No.8: Adsorption Kinetics-Pseudo second order reaction

Sl.No	Name of the adsorbent	Slope	Intercept	R^2
1	Dry leaf powder	0.048	0.5	0.923
2	Leaf ash	0.068	0.401	0.983
3	Dry stem	0.079	0.315	0.978
4	Stem ash	0.095	0.321	0.983
5	Dry flowers	0.104	0.203	0.985
6	Dry flowers ash	0.095	0.264	0.97
7	Dry fruit	0.96	0.289	0.983
8	Dry fruit ash	0.134	0.010	0.996

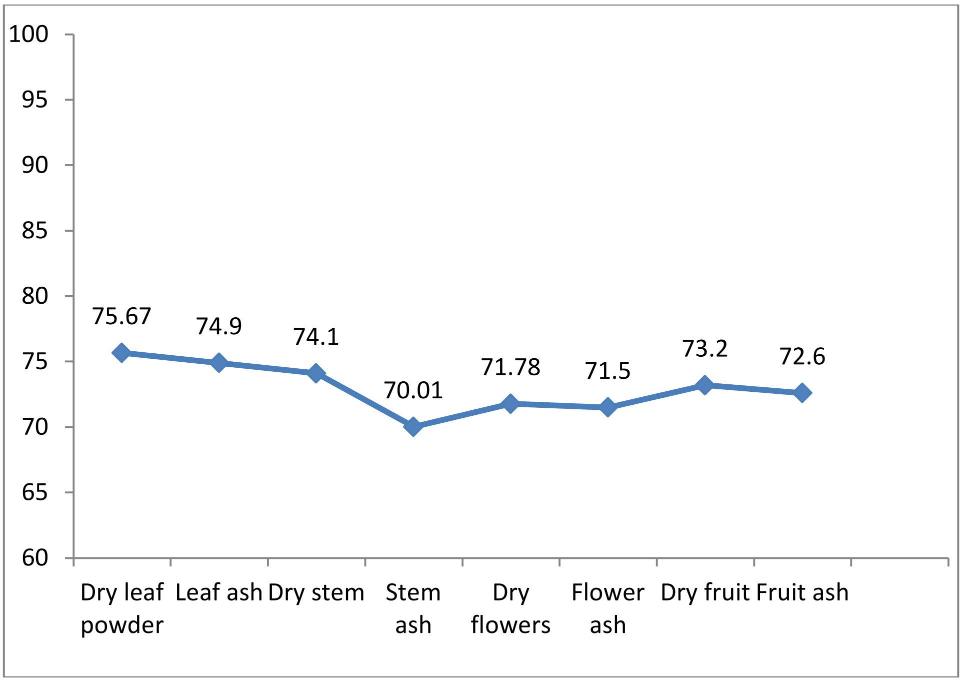
Application of this method

The polluted water is collected from paint industrial area near Vijayawada, Andhra Pradesh. The sample water is filtered with Wattmann filter paper and divided the water into equal aliquots in cleaned reagent bottles and 1 gram of each adsorbent of Clitoria Ternatea plant is added to the each bottle and kept for7 days with frequent shaking. By adopting the procedure in 2.1 the experiment was carried out. In that dry leaf powder has shown maximum adsorption of 75.67% and leaf ash shows 74.9%. The adsorption was shown by stem ash 70.01%. The % of removal of Lead by bio sorbent powders is presented in theTable-9. The absorption isotherm is shown in the Graph-9.

Table-9 Percentage of removal of Lead from industrial water

Sl.No	Name of the adsorbent	% of removal of Lead
1	Dry leaf powder	75.67
2	Leaf ash	74.9
3	Dry stem	74.1
4	Stem ash	70.01
5	Dry flowers	71.78
6	Dry flowers ash	71.5
7	Dry fruit	73.2

8	Dry fruit ash	72.6



Graph-9: % of removal of Lead from industrial water with various adsorbents

Conclusions

The bio-sorbents extracted from Clitoria Ternatea plant are used for the removal of Lead from polluted waters by developing a new and simple volumetric method. In this we find out the adsorption type and kinetic model. The percentage of removal of Lead is 85.01% with dry leaf powders is 78.94% with leaf ash, 78.94% with dry stem, 70.17% with stem ash, 73.68% with dry flowers and flower ash 77.19% with dry fruit and 73.68% with fruit ash at pH 6. The removal of Lead is observed maximum by most of the bio-sorbents. We observed it follows Longmuir adsorption method and pseudo first order method. The developed method is inexpensive and sustainable for the removal of Lead from polluted water which makes soil and water free Lead pollution.

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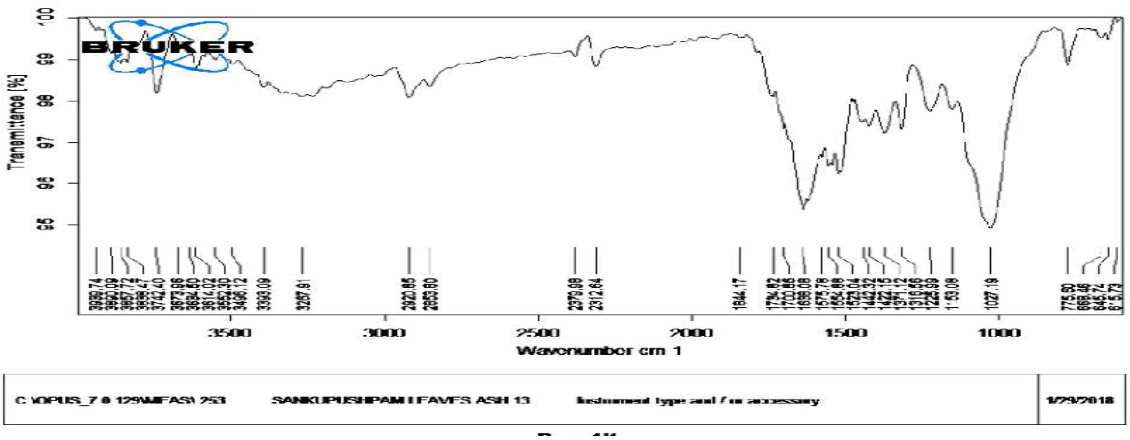

Fig-2.FT-IR spectra of Clitoria Ternatea Leaves Ash (before)

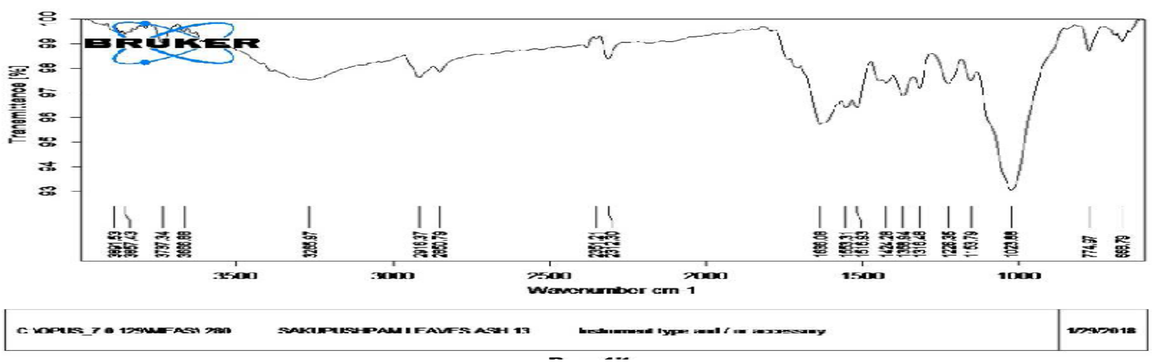

Fig-3. FT-IR spectra of Clitoria Ternatea Leaves ash (after)

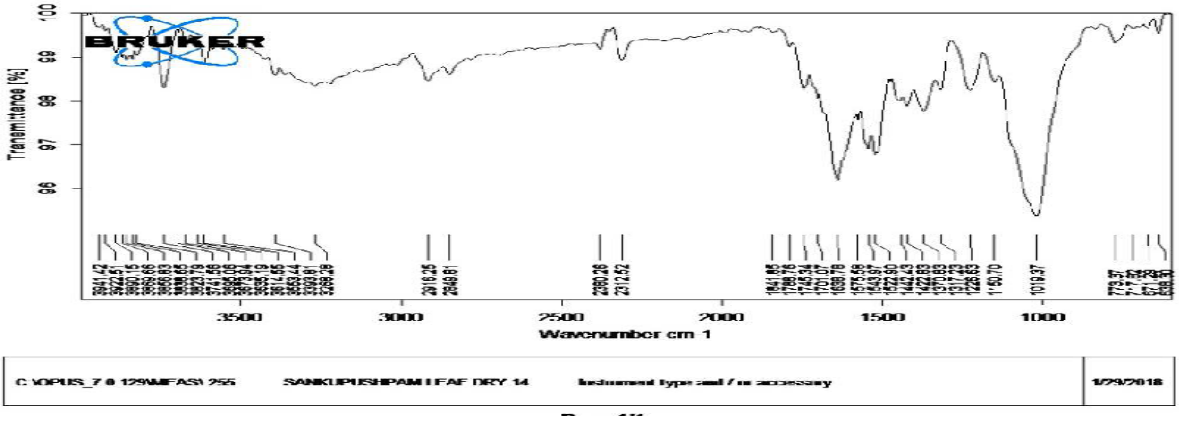

Fig- 4. FT-IR spectra of Clitoria Ternatea Leaves dry (before)

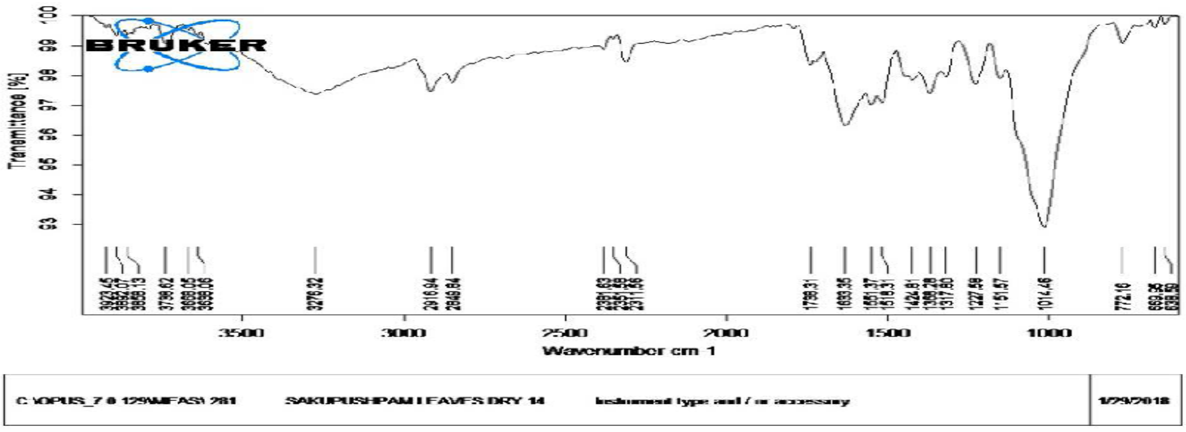

Fig-5. FT-IR spectra of Clitoria Ternatea Leaves dry (after)

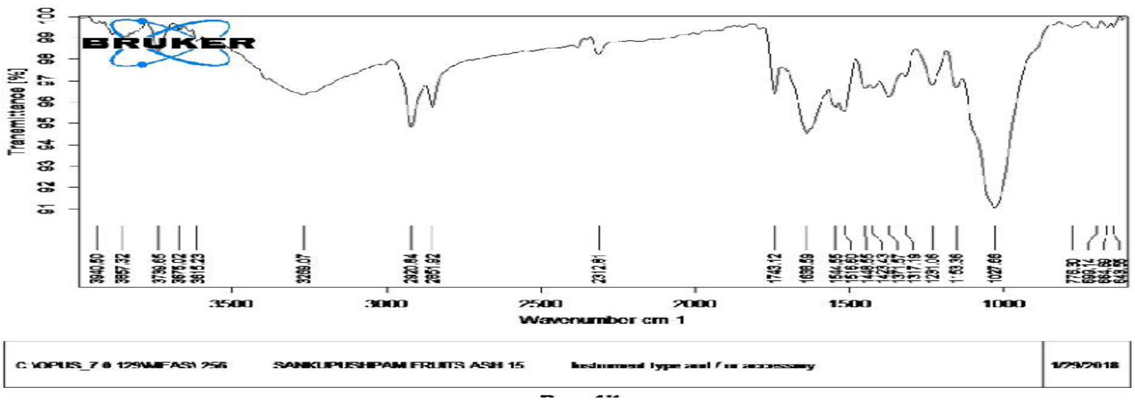

Fig-6. FT-IR spectra of Clitoria Ternatea fruits ash (before)

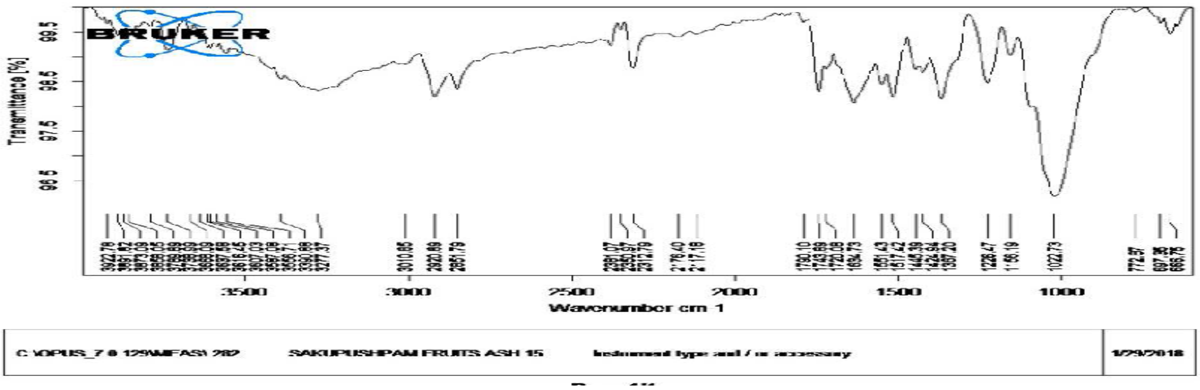

Fig-7. FT-IR spectra of Clitoria Ternatea Fruits Ash (after)

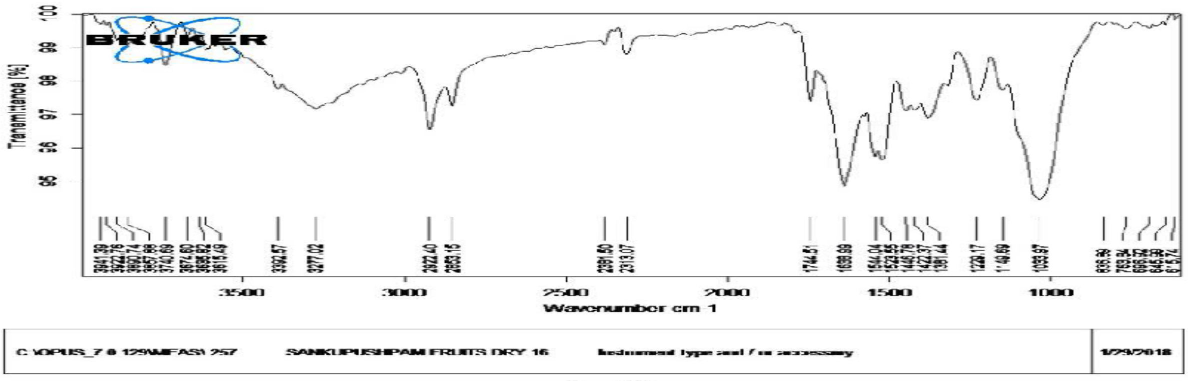

Fig-8. FT-IR spectra of Clitoria Ternatea Fruits Dry (before)

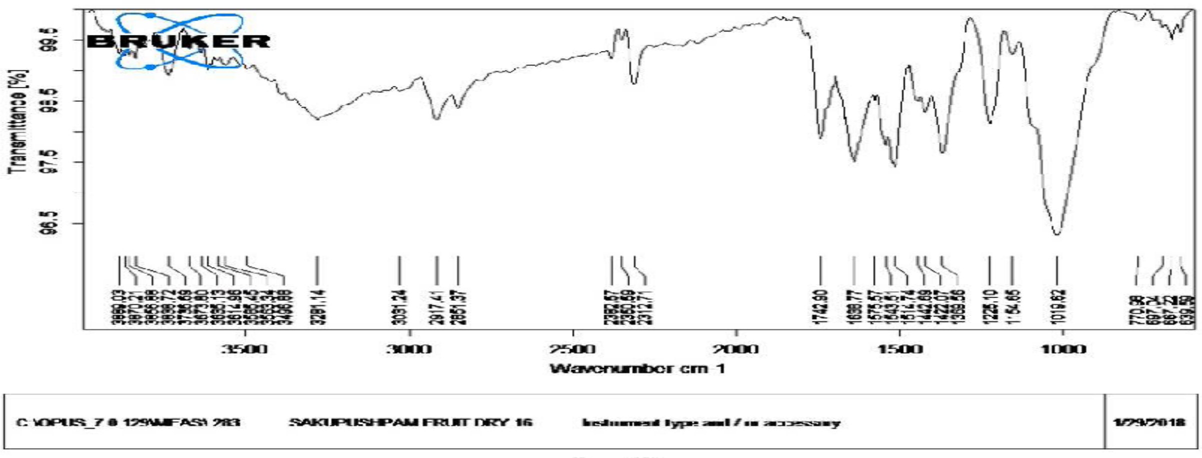

Fig-9. FT-IR spectra of Clitoria Ternatea Fruits Dry (after)

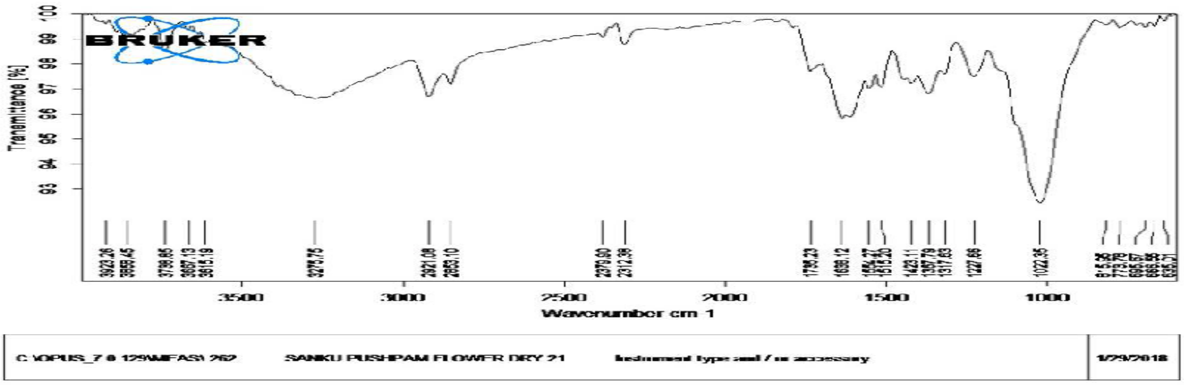

Fig-10. FT-IR spectra of Clitoria Ternatea flowers Dry (before)

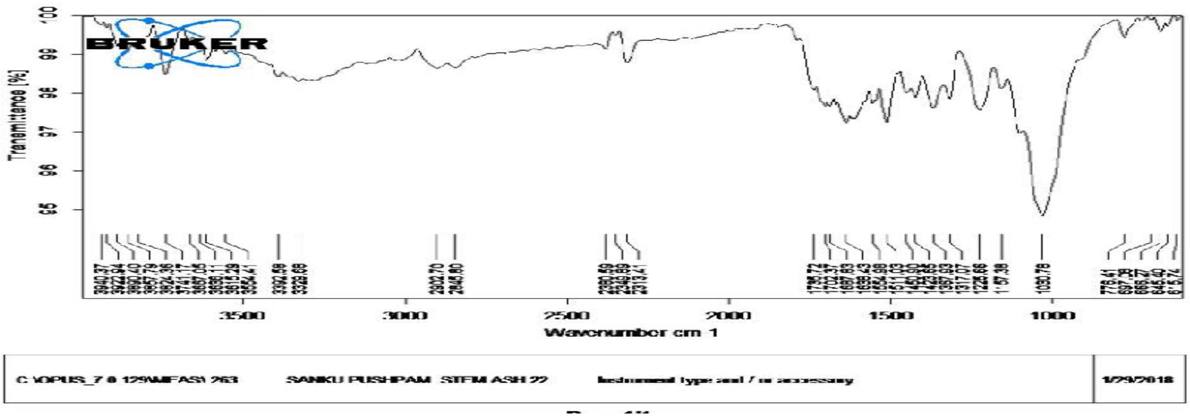

Fig-11. FT-IR spectra of Clitoria Ternatea Flower Ash (before)

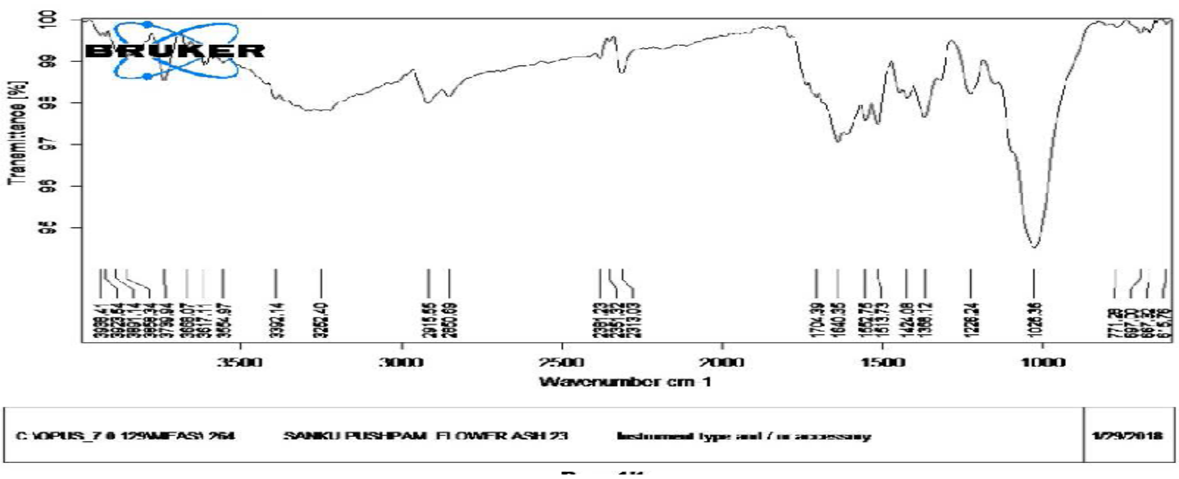

Fig-12. FT-IR spectra of Clitoria Ternatea stem ash (before)

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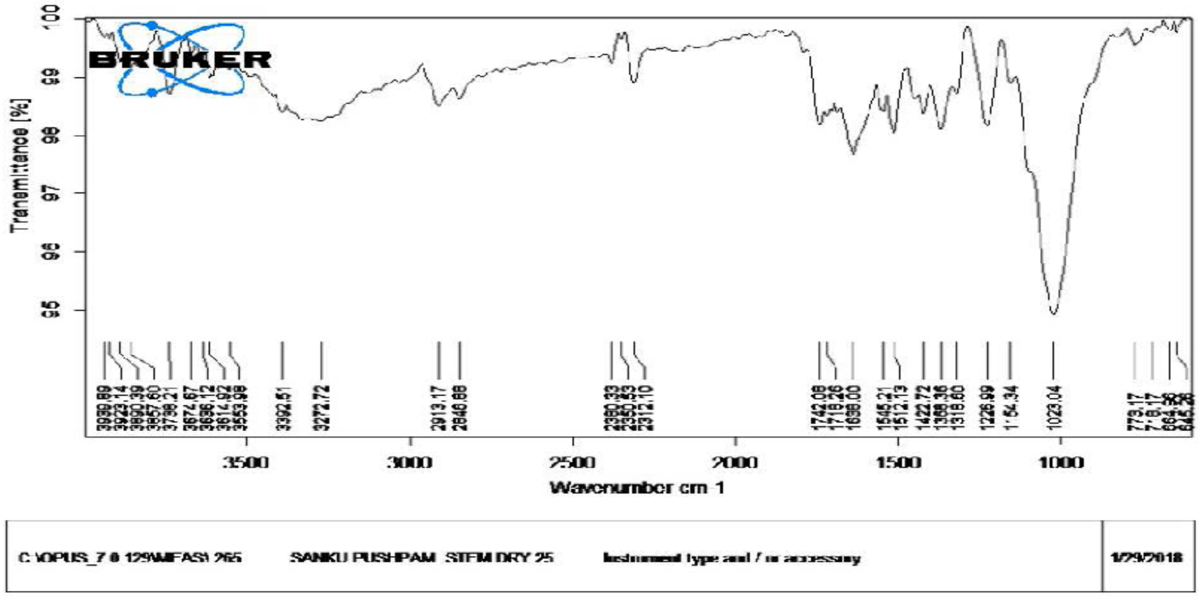

Fig-13. FT-IR spectra of Clitoria Ternatea Stem dry (before)