

**FUNCTIONALIZATION OF NOVEL BIO-ENCAPSULATED BEADS  
IN THE CHELATION OF NOXIOUS ANIONS AND FABRICATION  
OF FRP DEVICE AT LAUNDRY SITES – A GREENER APPROACH**

Thesis submitted to Bharathiar University for the award of the degree of

**DOCTOR OF PHILOSOPHY IN CHEMISTRY**

Submitted By

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Under the Guidance of

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PSGR Krishnammal College for Women



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**(K. VIVITHABHARATHI)**

*Abstract*

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## ABSTRACT

The unremitting industrial development has led to a subsequent increase in the amounts of wastewater generation. Fertilizer/ Laundry industries generate large volumes of wastewaters enriched with anions exceeding the standard limits, discharged into natural water bodies. Untreated disposal of these wastewaters pose serious threat to agricultural sector, aquatic life, human beings imposing adverse mutagenic/ carcinogenic effects. Removal of anions from these point sources poise as one of the major environmental concerns. Amidst, various methodologies tested for the reclamation of these anions, adsorption has been reported as a convenient method, due to many reasons such as flexible operation, specific toxicants' target and minimal generation of sludge. A number of zero cost and indigenous materials have been identified as promising sorbents to chelate the anions. The present study is focussed on the employment of *Camellia sinensis* stem (TCSS), *Elaeocarpus tectorius* seed (TETS), *Vicia faba* husk (TVFH) and *Gallus gallus domesticus* beaks (TGGDB), post relevant modifications as notable sequestrants for  $\text{PO}_4^{3-}$ ,  $\text{NO}_3^-$  and  $\text{SO}_4^{2-}$  ions from aqueous and laundry wastewater samples. These modified materials are subjected to physio-chemical parametric determinations and characterized distinctly by BET/ BJH, SEM, EDAX and FT-IR techniques to assess their precise nature, during pros and cons of the experimental setup. The factors influencing the adsorption capacities of the derived materials are experimentally verified by Batch mode and quantified through column setup for aqueous anionic media at laboratory levels. Initial and residual concentrations of the studied anions are complexometrically analysed using UV - Vis Spectrophotometer. Experimental data pertaining to Batch equilibration studies are statistically verified using SPSS software. A judicious comparison is made to assess the best sorbing ability among the four selected materials and the order of preferential adsorption between the three chosen anions. Desorption and regeneration experiments are performed for anion laden sorbents to enumerate their reusable property. Varied isothermal / kinetic models, dynamic behaviour of the verified systems is validated to understand the adsorption equilibrium and kinetic / thermodynamic behaviour. Calcium alginate, goethite and magnetite functionalized bio-beads are synthesized and characterized using XRD, TG-DTA and VSM methods. Sorption efficacies of these synthesized beads are recorded by

pilot studies and extended to column verification, followed by kinetic model validation. Exhausted column material is tested for its nutrient value in plant vegetation using phosphate solubilizing bacteria, thereby minimizing its load as a secondary pollutant. Based on the laboratory recordings, a prototype device is designed and installed at a laundry unit to promote the applicability of the novel material as an excellent sorbent at the field level.



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## *List of Abbreviations and Notations*

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## LIST OF ABBREVIATIONS AND NOTATIONS

|          |   |
|----------|---|
| CSS      | <i>Camellia sinensis</i> Stem                 |
| ETS      | <i>Eleocarpus tectorius</i> Seed              |
| VFH      | <i>Vicia faba</i> Husk                        |
| GGDB     | <i>Gallus gallus domesticus</i> Beaks         |
| CPCB     | Central Pollution Control Board               |
| TCSS     | Treated <i>Camellia sinensis</i> Stem         |
| TETS     | Treated <i>Eleocarpus tectorius</i> Seed      |
| TVFH     | Treated <i>Vicia faba</i> Husk                |
| TGGDB    | Treated <i>Gallus gallus domesticus</i> Beaks |
| SEM      | Scanning Electron Microscope                  |
| BET      | Bruner Emmett Teller                          |
| BJH      | Barrett Joyner Hammett                        |
| FT-IR    | Fourier Transform Infrared Spectrophotometer  |
| EDAX     | Energy Dispersive X-ray Spectrometer          |
| UV - Vis | Ultra Violet Visible Spectrophotometer        |
| VSM      | Vibrating Sample Magnetometer                 |
| $M_s$    | Saturation Magnetization                      |
| XRD      | X-Ray Diffraction                             |
| TGA      | Thermo Gravimetric Analysis                   |
| DTA      | Differential Thermal Analysis                 |
| ZPA      | Zeta- Potential Analyzer                      |
| PSA      | Particle Size Analyzer                        |
| Conc.    | Concentration                                 |

|                          |  |
|--------------------------|--|
| $\text{pH}_{\text{zpc}}$ | pH at which the surface charge of the adsorbent is zero                          |
| $q_e$                    | Amount of anions adsorbed per gram of the adsorbent (mg/g)                       |
| $C_i$                    | Initial anion concentration (mg/L)   |
| $C_e$                    | Equilibrium anion concentration in solution (mg/L)                               |
| $R^2$                    | Correlation Coefficient  |
| $q_e$                    | Amount of anions adsorbed per gram of adsorbent at equilibrium (mg/g)            |
| $q_t$                    | Amount of anions adsorbed per gram of adsorbent at time 't' (mg/g)               |
| $q_m$                    | Maximum monolayer adsorption capacity (mg/g)                                     |
| $B$                      | Langmuir Adsorption Constant   |
| $K_F$                    | Freundlich Adsorption Capacity (mg/ g)   |
| $N$                      | Freundlich Isotherm Constant   |
| $A_T$                    | Temkin Equilibrium Binding Constant  |
| $b_T$                    | Temkin Heat of Adsorption  |
| $R$                      | Gas Constant (8.314 J/mol K)   |
| $\beta_{DR}$             | Mean free energy of sorption per mole of adsorbate ( $\text{mol}^2/\text{J}^2$ ) |
| $\epsilon$               | Polanyi Potential  |
| $\epsilon$               | Mean Free Energy (kJ/mol)  |
| $k_1$                    | Pseudo First Order Adsorption Rate Constant ( $\text{min}^{-1}$ )                |
| $k_2$                    | Pseudo Second Order Adsorption Rate Constant (g/mg min)                          |
| SSE                      | Sum of Error Squares   |
| $\alpha$                 | Elovich Initial Adsorption Rate ( mg/g min)                                      |
| $\beta$                  | Elovich Adsorption Constant (g/mg)   |
| $K_i$                    | Intraparticle Rate Constant ( $\text{g}/\text{mg min}^{1/2}$ )                   |
| $\Delta G^\circ$         | Gibb's free energy change of adsorption (kJ/mol)                                 |

|                  |  |
|------------------|--|
| $\Delta H^\circ$ | Enthalpy change of adsorption (kJ/mol)           |
| $\Delta S^\circ$ | Entropy change of adsorption (J/mol K)           |
| $K_T$            | Thomas Constant (L/mg min)                       |
| $q_T$            | Adsorption capacity (mg/g)                       |
| $Q$              | Volumetric flow rate (mL/min)                    |
| $M$              | Mass of the adsorbent (g)                        |
| $C_0$            | Initial Concentration (mg/L)                     |
| $C$              | Effluent Concentration (mg/L)                    |
| $K_{AB}$         | Kinetic Constant (L / mg min)                    |
| $F$              | Flow rate (mL / min)                             |
| $Z$              | Bed depth (m)                                    |
| $N_0$            | Saturation constant (mg/L)                       |
| $t$              | time   |
| $C_0$            | Influent concentration (mg/L)                    |
| $C_i$            | Effluent concentration (mg/L)                    |
| $K_{YN}$         | Velocity constant (L / min)                      |
| $\tau$           | Time required for 50 % of adsorbate breakthrough |
| $t$              | Sampling time                                    |



*List of Instruments / Equipments used  
for Various Studies*

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## **LIST OF INSTRUMENTS / EQUIPMENTS USED FOR VARIOUS STUDIES**

1. UV – Visible Spectrophotometer
2. BET Surface Analyzer
3. CHNS Analyzer
4. Scanning Electron Microscope
5. Energy Dispersive X- ray Spectrometer
6. Fourier Transform Infrared Spectrophotometer
7. Ocular Micrometer
8. X-ray Diffractometer
9. Vibrating Sample Magnetometer
10. Thermo Gravimetric – Differential Thermal Analyzer
11. Thermostat Controlled Mechanical Shaker
12. Digital pH Meter