

**SYNTHESIS, CHARACTERIZATION OF CaO/C NANOCOMPOSITE
FROM DEXTROSE SOURCE MECHANISM FOR AZO DYES
REMOVAL FROM AQUEOUS SOLUTION**

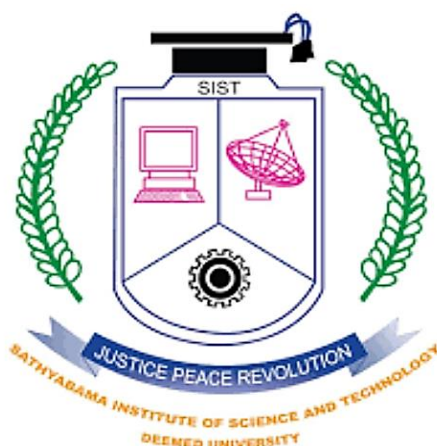
Submitted in partial fulfillment of the requirements for the award of

Bachelor of Technology degree in
Chemical Engineering
by

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**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with grade "A" by NAAC

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **KARTHIKA M (38190005), RASHEEQA M (38190010), SHEETAL CHANDRASEKARAN (38910014)** who carried out the project entitled **"SYNTHESIS, CHARACTERIZATION OF CaO/C NANOCOMPOSITE FROM DEXTROSE SOURCE MECHANISM FOR AZO DYES REMOVAL FROM AQUEOUS SOLUTION"** under my supervision from September 2021 to April 2022.

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DECLARATION

I **KARTHIKA M (38190005)** hereby declare that the Project Report entitled “**SYNTHESIS, CHARACTERIZATION OF CaO/C NANOCOMPOSITE FROM DEXTROSE SOURCE MECHANISM FOR AZO DYES REMOVAL FROM AQUEOUS SOLUTION**” done by me under the guidance of **Dr. S. SATHISH, M.E., Ph. D.**, and **Dr. D. KRITHIGA, M.Sc., Ph.D.**, at Sathyabama Institute of Science and Technology is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

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DECLARATION

I **SHEETAL CHANDRASEKARAN (38190014)** hereby declare that the Project Report entitled **“SYNTHESIS, CHARACTERIZATION OF CaO/C NANOCOMPOSITE FROM DEXTROSE SOURCE MECHANISM FOR AZO DYES REMOVAL FROM AQUEOUS SOLUTION”** done by me under the guidance of **Dr. S. SATHISH, M.E., Ph. D.,** and **Dr. D. KRITHIGA, M.Sc., Ph.D.,** at Sathyabama Institute of Science and Technology is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

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ABSTRACT

Several rising economies rely heavily on the textiles sector to drive their economic development. As a result, treating textile wastewater has become a global requirement. Despite the availability of numerous mechanisms for removing toxic pollutants from textile effluents, such as chemical oxidation, adsorption, biodegradation, ion-exchange, and photocatalytic processes, adsorption remains the most advantageous technique due to its low cost, simple design, high efficiency, and ability to be used in multiple ways. When it comes to adsorbents, carbonaceous adsorbents are unquestionably the most widely used to remove harmful contaminants in industrial effluents. A carbonaceous adsorbent CaO/C nanocomposite has been created and examined for its performance qualities in the elimination of azo dyes in this thesis research.

The carbon nanocomposite is synthesized using self-propagating solution combustion method. And the synthesized sample is characterized using SEM-EDS, FTIR, XRD, TGA and BET in order to find the elemental composition, structure, surface morphology, pore size, thermal stability. etc. Then, batch adsorption studies were performed to evaluate the optimum operating parameters where the optimum pH was found to be between 8-10, adsorbent dosage= 4g/l, temperature= 20°C to 30°C, concentration=150ppm and contact time= 60 minutes. Basic orange-2 was the azo dye used for the adsorption studies. The used adsorbent is again characterized to confirm whether the dye molecules are adsorbed by the adsorbent and to calculate the adsorbent efficiency followed by isotherm studies. The SEM image after adsorption confirms the closure of pores where the dye has been accumulated in the sites and EDS confirms the presence of the dye used. The adsorption isotherm was best described by Freundlich isotherm having the highest R^2 value of 0.963 and surface area was found to be $1.0188 \times 10^{+2} \text{ m}^2 \text{ g}^{-1}$. The mean pore diameter was found to be 1.9706 nm and the pore volume was found to be $5.0193 \times 10^{-2} \text{ cm}^3 \text{ g}^{-1}$.

Keywords: textile industry, adsorption, adsorbents, textile effluents, nanocomposite Adsorption studies, Freundlich isotherm, azo dye

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CHAPTER 1

INTRODUCTION

1.1 TEXTILE EFFLUENT IN WATER POLLUTION

Freshwater one of the most important elements on our planet, accounting for 70% of its surface area. Increasing industry and urbanization as a result of rising population contribute significantly to water pollution, which has negative consequences for human life, the environment, and the economy. Industries damage water sources with a variety of pollutants, including heavy metals, trace organic pollutants, organically bound metals and metalloids, inorganic species, pesticides, and detergents, among others. Taking a closer look at the textile business, it accounts for one-fifth of all industrial water contamination worldwide. The textile effluent is extensively polluted with pollutants such as colors, dissolved solids, suspended particles, and hazardous metals that are difficult to degrade with standard treatment procedures, and it inhibits sunlight penetration, which is essential for aquatic life. Textile effluents also result in greater levels of total dissolved solids, chemical oxygen demand, pH and biological oxygen demand. With the rising threat of water pollution, waste water treatment has become one of the fastest-growing sectors, with a slew of new research and studies attempting to balance the amount of water contaminated vs water cleansed and recovered for reuse. Every day, new technologies are developed, and some of the most widely utilized and effective strategies are mentioned here. The treatment of textile effluent by adsorption utilizing several innovative adsorbents is described in depth, with those adsorbents being classified as Carbon-based adsorbents, Nano adsorbents, Bio based adsorbents, Magnetic adsorbents, and Enzymatic adsorbents.

1.2 TEXTILE EFFLUENT TREATMENT METHODS

1.2.1 BIOLOGICAL METHODS

Aerobic, anaerobic, anoxic, facultative, or perhaps a hybrid of these biological techniques can be used. Throughout the presence of sufficient dissolved oxygen, anaerobic methods use microorganisms to detox the toxins from wastewater, whilst anaerobic techniques use bacteria lacking oxygen to remove contaminants from wastewater. Ecologically responsible, minimal, low-infrastructure, and close to the bottom costs, low sewage sludge, complete mineralization into non-toxic end products, and so on are all features of biodegradation beyond physical and chemical processes. The microorganisms employed and enzyme activity impact the efficacy of the breakdown of organic matter. As a response, countless microorganisms and enzymes have been produced and tested for dye degradation. Isolation of prospective microorganisms of their use for removal is a biologically important component of textile wastewater treatment. Colors in textile effluent water are eliminated by numerous microorganisms such as bacteria, fungi, and algae.

1.2.1.1 AEROBIC PROCESSES

Bacteria and microorganisms consume organic compounds, causing colloidal materials to agglomerate and flocculate, dissolved organic matter to oxidise to CO₂, and nitrogenous organic matter to degrade to ammonia, which is then metabolized to nitrite and then nitrate.

1.2.1.2 ANAEROBIC PROCESSES

Sludge digestion is mostly treated anaerobically by keeping the sludge at 350 degrees Fahrenheit and a pH of 7-8 for 30 days in a sludge digester. As end products, CH₄, CO₂, and some NH₃ are obtained.

1.2.2 CHEMICAL METHODS

1.2.2.1 ADVANCED OXIDATION PROCESS (AOPs)

The basic aim of AOP is to create and utilize hydroxyl free radical as a powerful oxidant to eliminate textile effluent molecules that cannot be oxidized by traditional oxidants.

1.2.2.1.1 Photocatalysis: Multiple irradiating solid photocatalysts are used in heterogeneous photocatalytic processes to keep generating radicals. Photodetectors can efficiently be photo-excited, resulting in the formation of electron donor and acceptor sites that initiate with the excitation of photon processes. Photo-excitation processes exist whenever ingested UV radicals have a potential ($h\nu$) equivalent to or larger than the semiconductor energy band gap (between the conductive and reactive bands). In the conduction as well as the valence bands, combination of electrons (e^-) and holes (h^+) are created which can either recombine or travel to the semiconductor (S) surface, where they react with chemical species adsorbed on the surface. $HO^\bullet$ is formed through a process of water ionization, oxygen ionosorption, and superoxide protonation processes.

The following aspects are the features of photocatalytic activity for dye removal from fabrics:

1. catalysts could be used in a variety of acoustic and mild situations
2. photolysis robust catalysts are easily obtainable internationally
3. the repair process can hypothetically use light from the sun as a spotless and cost-effective photo-source.

The foregoing is also some of the shortcomings of photo-catalytic degradation for fabric removal efficiency:

1. wasteful light exploitation and low $HO^\bullet$ emission proportionate
2. mass transfer barriers to the edge of the fixed motivator on a sample
3. high photoelectron and hole pair charge recombination

1.2.2.1.2 Fenton reagent: When Fe^{2+} and H_2O_2 are mixed under acidic circumstances, the traditional Fenton reaction produces a considerable quantity of $\text{HO}^\bullet$. In the presence of sufficient H_2O_2 , Fe^{2+} easily oxidises to Fe^{3+} in a couple of minutes. Due to the regeneration of Fe^{2+} derived from the reduction of Fe^{3+} , the reaction spreads quickly. The conventional Fenton procedure has the benefit of requiring no external energy and using very inexpensive chemicals. As a result, it may efficiently and inexpensively remove stubborn organic pigments from textile effluent. Furthermore, The Fenton process is straightforward and customizable to adopt in full-scale installations so it operates at ambient temperature and air altitude. Fenton-based AOPs have a limited pH range, which is one of their biggest drawbacks. The conventional Fenton reaction is confined to a 2–4 acidic pH stretches, whereas industrial textile effluent is often a basic solution.

1.2.2.1.3 Ozonation: Depending on the pH, ozonation can destroy textile colors in two ways. Under acidic circumstances, O_3 has the potential to operate as an electrophile on aldehydes, carboxylic acids, and other raw material for the production are formed by electron-rich functional groups in fabric dye complexes such as amines, aromatic rings and double bonds. O_3 has the ability to eliminate a huge spectrum of chemical compounds from the effluent from industry due to its high oxidation potential of 2.07 V. By a series of complicated chain reactions, O_3 quickly interacts with the combination of OH and produces other radical species, including such $\text{HO}^\bullet$ at basic pH. The excellent decolorization and biodegradability of O_3 -based AOPs are their key benefits. O_3 is excellent in dye wastewater photo-degradation because it attacks conjugated double bonds, which is very common in dye wastewaters.

1.2.2.2 CHEMICAL OXIDATION

Electrochemical oxidation is a high-efficiency, low-cost method for removing acid dyes, metal complexes, and dispersion colors, particularly for those comprising acid dyes, metal complexes, and disperse dyes. It's a low-temperature procedure that doesn't necessitate the use of any extra chemicals. An electrolytic cell that employs electrical energy to effect a chemical change is used in electrochemical oxidation. It has a positive anode for oxidation and a negative cathode for reduction, both of which are submerged in an electrolyte-like salt. Because dye wastewater has high

salt concentrations, it is an appropriate conducting medium. Several complicated processes take place at the anode, resulting in hypochlorite ions and OH free radicals, which destroy the dye compounds. Platinum, cobalt, palladium, copper, nickel, iridium, or other materials might be used as the anode. Chloride ions are oxidized to chlorine and ultimately hypochlorous ions (OCl^-) at the anode, whereas water molecules are oxidized to OH^- at the cathode. COD levels are reduced by 80 to 96 percent on average. When the salt concentration reaches 0.1M, COD elimination is 100 percent. Because of the increased chloride concentration and mass transfer to the anode, hypochlorite ions are produced, which can oxidize more organic compounds. Current density of 20 mA/cm^2 is optimal. 3.25–6.0 kWh/kg is the energy consumption reported. The ideal pH level is 4. COD elimination reduces when the pH rises to alkaline levels. This indicates that following therapy, pH regulation is essential.

1.2.3 PHYSIOCHEMICAL METHOD

1.2.3.1 COAGULATION

The sample of textile effluent (100 liters) is taken directly from textiles in new plastic containers that have been previously rinsed with dil. HNO_3 . Following collection, a few cc of concentrated HNO_3 is added to the effluent to inhibit microbial development and the container is shut to avoid air oxidation. Within a week of collecting the sample, the raw effluent is examined for physicochemical parameters such as pH, COD, TSS, TDS, turbidity, and so on. Then, in batch tests, a 200ml sample is split into five beakers and 25 percent acetic acid is injected to get the pH to 7. Then, using a graduated pipette and a glass rod, 1,2,3,4 and 5 ml of coagulants such as PAC, FeCl_3 , AlCl_3 , $\text{Fe}_2(\text{SO}_4)_3$ are added to all beakers. Quick coagulation and sedimentation will occur as a result of the addition. The coagulation and sedimentation appear to be finished in 30 minutes, but the system must be left undisturbed for roughly 6 hours for the process to be completed. The supernatant liquid is then carefully decanted from the treated effluent for examination of effluent quality parameters. Coagulation does have its own limitations. firstly, due to reduced decolorization capability and the massive number of by-products developed by sludge production. Because the sludge from the treatment process contains soil

conditioning properties, it may be utilized as a component of a slow release organic fertilizer. Though heavy metals attached to organic molecules in sludge may provide a rich nutrition supply, prolonged usage may raise heavy metal concentrations in soil. These technologies also work poorly when reactive and vat dyes are present in the water.

1.2.3.2 ION EXCHANGE

Cation and anion removal is achieved from drainage using this approach. Synthetic resins are often exploited in this methodology. In the softening of hard water, the ion exchange process is increasingly employed. It has only been used to neutralize pigment from water, however. The benefit of this strategy would be that no adsorbents are destroyed. It could be used to get traces of water-soluble hues. It is unsuitable, however, for water-insoluble colourant like scattering dyes. It entails passing waste textile effluent through ion exchange resin beds, where part of the waste effluent's unwanted cations and anions are exchanged for sodium or hydrogen ions in the resins.

1.2.3.3 FILTRATION

To remove pollutants from textile effluents, filtering approaches such as reverse osmosis, ultrafiltration, nanofiltration as well as the microfiltration (MF) have been deployed. For removal technologies, filter media qualifying criteria, as well as their ability to consider temperature and chemical content of wastewater containing, are crucial. In textile manufacturers, membrane technologies are developed to decrease BOD, COD, and colour from effluent wastewater, as well as retrieve salt and reduce the waste volume. Membrane filtering has the advantages of being a rapid technique with a small footprint and the ability to reuse the material. Investment made costs, membrane clogging, pollutants including relatively insoluble hues (for illustration, indigo dye), and pectin dye degradation membranes, on the other hand, all have significant downsides that demand alternative care. A paper filter was employed as a pre-treatment for the membrane filtration in order to minimize membrane fouling, which would drastically reduce the membrane's lifetime. The suspended particles in the textile effluent were eliminated using this pretreatment. Although the microfiltration did not provide acceptable permeate

quality, it may be utilized as a pre-treatment for nanofiltration or a multistage can be employed to increase the MF output. Ultrafiltration removes macromolecules and particles, however contaminating chemicals like dyes are never completely removed; the removal rate varies between 31 and 76 percent. Colored effluents from the textile sector are treated by nanofiltration. Nanofiltration, in addition to adsorption, can be utilized to cleanse textile dye effluents. Reverse osmosis provides a high-quality permeate with a retention value of 90% or greater. RO may be used to discolor dye house waste water and remove chemical auxiliaries, mineral salts, and hydrolyzed reactive dyes.

Table 1.1 Different types of filtration

Process	Pore size (micron)	Molecular weight	Removed substance
MF	0.007-2.00	>100000	Bacteria, pigments, oil. Etc.
UF	0.002-0.10	1000-200000	Colloids, virus, protein. etc.
NF	0.001-0.07	180-15000	Dyes, pesticides, divalent, ions. etc.
RO	<0.001	<200	Salts and ions

1.2.3.4 ADSORPTION

After studying 13 textile enterprises, Owen (1978) found that granular activated carbon adsorption has emerged as a feasible and cost-effective method for removing colour from textile effluents. In commercial applications, activated carbon

(AC) is the most often utilised adsorption material. It possesses a high surface area to volume ratio, as well as mechanical strength to endure attrition, acidic stability, and adaptability. Activated carbon is made using coconut shell charcoal or coal. Coconut shell charcoal is frequently used for textile dye effluent. AC is available in two forms: granular (GAC) and powder (PAC). GAC is made up of irregularly shaped particles that have been milled and sieved. The sizes of these items range from 0.2 to 5mm. They are tougher and endure longer than PAC, are easier to handle, filter huge amounts of gas or liquids of constant quality, and can be reactivated and reused several times. The particle size of PACs is usually rather small. Distribution ranging from 5 to 150Å, although coarser and finer grades are available. The advantages of PAC include its decreased processing costs and operational flexibility. As process conditions change, the dose of PAC may readily be raised or lowered. They are introduced to the liquid to be treated, mixed with it, and then removed by sedimentation and filtering following adsorption. In general, the more interior surface area a carbon has, the more effective it is. The contact angle of AC fluctuates from 500 to 1500m²/g. To achieve the large surface area, steam is used to activate the charcoal. The huge surface area allows a large number of contaminating molecules to accumulate. The rhetoric surrounding of a GAC to capture complex substances is dictated by molecular surface tension, total surface area usable per unit weight of carbon, and the level of contaminants in the wastewater stream. It's widely used as a polishing step for textile wastewater treatment after physicochemical or secondary biological treatment to adsorb tiny amounts of colors. The technique is simple to use, has a small footprint, and is based on established technology. The photo-degradation efficiency of activated charcoal is influenced by the temperature of the sewerage, the pore volume of the activated charcoal, the initial concentration, the temperature, the solution pH, and the dye variety. Because activated carbon surfaces are amphoteric, the pH of the solution influences dye adsorption. Cationic dyes adsorb better at higher pHs, whereas anionic dyes adsorb better at lower pHs. GAC, in particular, may adsorb dyes, sulfides, heavy metals, and nitrogen compounds over a wide pH range. The term "Empty Bed Contact Time" refers to the time spent in contact with an empty bed (EBCT). The EBCT is determined by multiplying the AC bed's total capacity by the fluid flow velocity. For maximum effectiveness, an EBCT of 10–15 minutes and a temperature of 30–400°C is commonly advised. For cationic, mordant, and acid

dyes, activated carbon achieves high clearance rates of over 90%. The removal effectiveness of direct, diffused, reactive, and Sulphur dyes is modest at 40%. Because of their limited solubility and colloidal characteristics, insoluble dyes like vat, dispersion dyes, and pigments are difficult to adsorbed by activated carbon. For dye removal rates of 60–90%, activated carbon consumption is typically approximately 0.5–1.0kg/m³ effluent. The most significant drawback of activated carbon is its expensive price. Because dye adsorption is not compound-specific, it competes with other organic compounds. Without pretreatment, suspended particles would clog activation sites, necessitating the use of huge amounts of activated carbon, making the procedure costly. As a result, suspended solids should not exceed 20 mg/L. When one unit gets depleted, the other is usually utilized in series. It is necessary to regenerate the activated carbon. Even yet, roughly 10% to 15% of it is lost.

1.1 AZO DYE- BASIC ORANGE 2 (Chrysoidine Y)

Basic orange 2 is an azo compound. And it is red brown powder. It is soluble in water, slightly soluble in acetone and insoluble in benzene. It is mainly used in the dyeing of silk, wool, acrylic and cotton. It is yellow-orange soluble in water, and it is also soluble in ethanol. It is yellow soluble in strong sulfuric acid, and it turns into orange after diluted. It is orange soluble in nitric acid. The Structure of the dye is given in the fig 1.1.

In this study, synthetic Basic Orange 2 (basic azo-dye which is also known as Chrysoidine Y) was chosen as an example of cationic dyes in aqueous solutions. BO2 is one of the azo-dyes used widely in the textile industries and has toxicity to humans through oral or skin administration.

Molecular formula: $C_{12}H_{13}ClN_4$

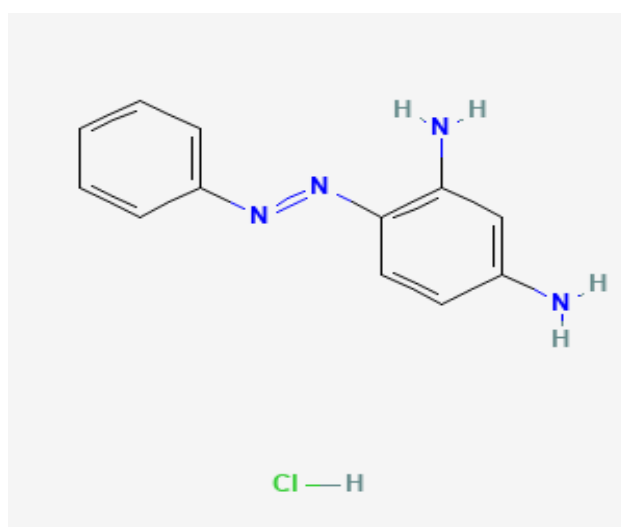


Fig 1.1 Structure of Basic Orange 2

1.3.1 IMPACTS OF BASIC ORANGE 2

1. Generally, dyes as organic pollutants have a synthetic origin and are characterized by complex aromatic molecular structures and high physicochemical, thermal, and optical stability.

2. Basic dyes are cationic dyes and are widely used in various industries. These dyes are commonly applied in the paper, pharmaceutical industries, and dyeing of polyester and nylon fibers in textile industries. Because of these features, dye-containing contaminations cause many environmental severe problems according to their low biodegradability characteristics, and toxicity to aquatic life, and the conventional biological treatment procedures are not efficient enough to treat wastewaters.
3. Dyes can damage the body organs due to their carcinogenic and mutagenic effects on humans and other organisms depending on dose and duration of exposure
4. Therefore, uncontrolled discharge of the coloured water and solutions containing dyes and toxic heavy metals from industries produce considerable polluted waters and with due attention to the potentially harmful effects of such compounds on the environment and humans, they must be removed from wastewaters before discharging into the surrounding environment
5. Adsorption was found as a promising way for the hazardous pollutants removal from the wastewaters due to the advantages of its simple operation, effectiveness, high treatment efficiency, and applicability for the removal of various types of pollutants such as dyes and heavy metals.

1.3.2 USES OF BASIC ORANGE 2

1. Basic orange 2 is mainly used to dye silk, wool, acrylic fiber, leather, and cotton. It is also used in the dyeing of paper, feather, plant and bamboo fiber.
2. Used for silk, wool, acrylic, tannin mordant dyeing cotton dyeing and printing, direct discharge printing. In addition, also used in leather, paper, feathers, plants and bamboo products dyeing.

CHAPTER-2

LITERATURE SURVEY

CARBONACEOUS ADSORBENTS OBTAINED FROM WASTES USED FOR TEXTILE EFFLUENT TREATMENT

Adsorption of Dyes onto Carbonaceous Materials Produced from Coffee Grounds by Microwave Treatment

(Mizuho Hirata, Naohito Kawasaki, Takeo Nakamura, Kazuoki Matsumoto, Mineaki Kabayama, Takamichi Tamura, and Seiki Tanada)

Production of carbonaceous materials from coffee grounds are done by:

Carbonation by muffle furnace: Finest coffee granules (CGR) were processed at 383 degrees Fahrenheit for 24 hours in a day, after that, it was deposited in a enclosed compartment that also was immersed into more of an electric arc furnace that was elevated to 1073 degrees Fahrenheit at a rate of 10 degrees Fahrenheit per minute in a N₂ gaseous phase, maintained for 1 hour at this temperature, The carbonaceous matter was therefore chilled (CGM).

Carbonation using microwave: Slippery coffee granules were microwaved and carbonized in airflow for 7 minutes (CG7), 9 minutes (CG9), and 12 minutes (CG12) at a frequency of 2450 MHz with an output power of 500 W. (CG12). Carbonation acquired a temperature of over 673 K.

The carbonaceous material's elemental makeup and surface area are discovered. Because a portion of the carbon in coffee grinds became CO₂, the output of carbonaceous material was observed to decrease as the microwave treatment time increased. The amount of carbon in CG9 was reported to be greater than in CGR attributed to carbonization and reduced than those in CGM since this temperature in the electric furnace was higher than in the microwave. 3750 and 75 Wh of power are used in the electric furnace and microwave treatment, respectively. The dyes utilised in the adsorption investigations were methylene blue, Orange II, and gentian violet. The proportion of dyes eliminated increases as the microwave treatment time increases. Methylene blue dye solution and gentian violet (basic dyes) were

extracted from all the observations. While removing the orange II (acidic coloring) needed a lengthier microwave treatment duration. Because the synthetic carbonation ingredient has a higher base consumption, this is the case. As a result, it was discovered that the material's acid and basic intake plays a significant impact in dye adsorption. Microwave treatment is shown to be more environmentally friendly since it uses less power and produces less CO₂. In the year 2000, the world's yearly coffee bean consumption was estimated to be at 6,432,000 tonnes, producing 3,460,000 tonnes of CO₂ when burned. However, in this case, CO₂ is only formed when coal is carbonized in an electric furnace. This is a fantastic concept because it cuts CO₂ emissions to around 1,370,000 and 1,112,000 pounds per year, respectively.

Sugarcane bagasse and its potential use for the textile effluent treatment

(Juan Carlos Cueva-Orjuelaa, Angelina Hormaza-Anaguanob & Andrés Merino-Restrepob)

Sugarcane bagasse SCB, a high-cellulose, hemicellulose, and lignin agricultural by-product, was purchased from local markets, to eliminate organic complexes, it was rinsed with deionized water and 2.0 percent hydrogen peroxide and dried for 48 hours at 100°C. The SCB is next pretreated and mesh sieve to a particle size range of 0.3-1.0 mm. The adsorbent featuring these capabilities was retained in containers for any further research. Some early research was done to see how pH and particle size affected the removal of BR46 from SCB. The pH of the solution was found to be between 2.0 and 11.0, with a having a detrimental between 5.0 and 7.0. Particle sizes were specified in the bands of 0.3-0.5 mm, 0.5-0.7 mm, and 0.7-1.0 mm, with the 0.3-0.7 mm showing superior adsorption. Adsorption studies were carried out with the optimum settings, and the percentage of removal was estimated by,

$$\% \text{ Removal} = \left[\frac{C_o - C_f}{C_o} \right] * 100$$

Where C_o and C_f are initial and final concentration of the dye.

The findings showed that the agricultural by-product SCB may be used as a nontraditional adsorbent for removing BR46 from aqueous solution. This feature

highlights SCB's new method to dye effluent decontamination that is aimed at improving the environment.

Rice Husk Based Adsorbents for Textile Effluent Treatment

(G.K. Jayathunga and B.M.W.P.K. Amarasinghe)

Rice husk (RH) was gathered from neighboring rice fields and washed with water until the water was practically colorless, removing soluble and coloured components. The RH is macerated and preserved in airtight polythene containers until employed after already being scrubbed with distilled water and oven solidified for 12 hours at 85 degrees Celsius. After that, the washed RH is crushed and sieved. Small has adsorption capacity, according to preliminary testing, thus the size is kept at 500-710 μ m. After that, it's impregnated in 0.1 M NaOH solutions along with 0.1 M HCl for 6 hours before being washed and dried in the oven. The RH is then chemically treated by impregnating it in phosphoric acid solutions containing 60% phosphoric acid. It is then carbonized and thermally processed at 700°C in a muffle furnace.

Textile dyes solutions were obtained by dissolving basic green 4 and acid blue 80 in distilled water and thereafter testing the RH adsorbent in authentic textile water. The adsorption tests were carried out in a fixed bed adsorption column after the optimum parameters were identified. To obtain the desired bed height, a known weight of adsorbent was poured in a 3 cm diameter glass column. A dye sample solution 30 mg/L dye was injected into the gradient at a uniform velocity of 20 ml/min through the surface using only a peristaltic pump. The solution departing the bottom of the column was compiled and analyzed using a UV visible spectrophotometer at various time intervals. It was discovered that when the adsorbent was dried, the adsorption sites remained unoccupied, thus it was pre-wetted before use. The dyes were also successfully removed, with thermally treated RH performing similarly well as commercial activated carbon.

Preparation of carbonaceous materials from pyrolysis of chicken bones and its application for fuchsine adsorption

(Letícia Nascimento Côrtes & Susanne Pedroso Druzian & Angélica Fátima Mantelli Streit & Tito Roberto Sant'anna Cadaval Junior & Gabriela Carvalho Collazzo & Guilherme Luiz Dotto)

Chicken bones (CB) were procured from small stores and employed as a biochar and activated carbon synthesis precursor material. The CB precursor was disinfected many rinsed with deionized water before becoming oven cured at 60 degrees Celsius for 48 hours. It was then milled into particles smaller than 2 mm in a blade miller.

Chicken bones were pyrolyzed to produce CB biochar. The pyrolysis studies have been performed in a laboratory reactor in a batch configuration. The reactor seems to have a length of 90 to 99 cm, an outside diameter of 49 millimeter, and an internal diameter of 43 mm. The dimension of the oven that can be used approximately 516 mm. Two resistors, each rated at 1900 W, were used to heat the reactor electrically. Two K-type heat exchangers have been put inside the furnace. The system is elevated at $10\text{ }^{\circ}\text{C min}^{-1}$ till $800\text{ }^{\circ}\text{C}$ underneath a 150 mL min^{-1} N_2 flow rate, and the reactor was supplied with a fixed quantity of CB. After exceeding the final temperature (800°C), the system remained isothermal for 60 minutes. The reactor then was refrigerated with nitrogen until it attained a steady temperature after which the solid that had formed was removed, measured, and stored for later study. This material was given the authorized CB biochar.

CO_2 activation maintained the thermal degradation of chicken bones, resulting in CB-activated carbon. The apparatus were elevated at $10\text{ }^{\circ}\text{C min}^{-1}$ till $800\text{ }^{\circ}\text{C}$ under a 150 mL min^{-1} N_2 flow rate, and the reactor was loaded with a set proportion of CB. The apparatus remained isothermal for 60 minutes after reaching the maximum temperature (800°C). The apparatus were then elevated to 950°C followed by storage at that atmosphere of 60 minutes with a CO_2 flow of 150 mL min^{-1} . CB-activated carbon was the substance that formed from the activation. A thermostatic shaker was used to execute adsorption trials using fuchsine dye as just an impurity, followed by centrifugation at 4000 rpm for 15 minutes to differentiate the solid phase from the residual solution. Fuchsine in the liquid state was measured spectrophotometrically and a spectrophotometric approach at the maximum

wavelength absorbance (540 nm). The removal percentage was computed as follows:

$$\% \text{ Removal} = \left[\frac{C_o - C_f}{C_o} \right] * 100$$

Where C_o and C_f are initial and final concentration of fuchsine dye.

CB-activated carbon was shown to be more effective than CB biochar in eliminating colour in a synthetic textile effluent, demonstrating that alternate carbonaceous substances from chicken bones might be used to treat coloured wastewater.

Use of Industrial Coal Waste Materials as Adsorbents for Textile Effluent Remediation

(Mohamed H. Mohamed, Marissa Pirlot, Michael K. Danquah, Lee D. Wilson)

After that, a mining ore mixture was made by swirling a mixture of mining ore powder (45wt%), propyl gallate (as just an adherent, 5wt%), and distilled water for 10 hours (50wt percent). After passing through a centrifugal atomizing disc, the slurry was heated to 200°C and microbeads were formed in the evaporation cascade. To manufacture porcelain micro, the beads were melded at 900°C and then chilled. This preparation procedure is easy and economical for mass manufacturing of ceramic adsorbents. They were also discovered to be one-way adsorbents since renewing them was prohibitively expensive.

Ore activated carbons being mixed using 100ml CR or CB solutions in a 250ml flask. This vial then was enclosed and spun at 200rpm and 25°C for 3 hours. After centrifuging the material out from precipitate for 5 minutes, the dye intensity was calculated using UV spectrophotometer. This removal percentage were also determined. At pH=8, cationic blue was removed 99.7% of the time, whereas cationic red was removed almost 100% of the time at pH=12. More research is needed to enhance the adsorbed quantity and increase the surface area of the ceramics. In addition, a preliminary hazard investigation for the landfilling including used earthenware must be carried.

CHAPTER - 3

AIM AND SCOPE

3.1 AIM

The important aim is to synthesize and characterize CaO/C nanocomposite and study its application.

3.2 SCOPE

The main scope of this project is to evaluate the obtained CaO/C nanocomposite to investigate its properties such as composition, structure, and morphology, and investigate its utilization in the degradation in the selected Azo industrial fabric colouring from the aqueous solution.

CHAPTER – 4

MATERIAL AND METHODOLOGY

4.1 MATERIALS

The list of materials employed to synthesize CaO/C nanocomposite are listed below:

4.1.1 CHEMICALS REQUIRED

- Calcium carbonate salt
- Dextrose
- Distilled water
- Azo dye

4.1.2 APPARATUS REQUIRED

- Beaker
- Standard flask
- Glass rod
- Crucible
- Timer
- Mortar and Pistol

4.1.3 EQUIPMENTS REQUIRED

- Muffle furnace
- Hot air oven
- Hot plate magnetic stirrer
- UV Spectrophotometer

4.2 METHODOLOGY

The methodology used to synthesize CaO/C nanocomposite is by self-propagating solution combustion method.

4.2.1 PREPARATION OF CaCO_3 AND DEXTROSE SOLUTION

In a 100ml standard flask, dissolve 36 grams of dextrose by using the deionized double distilled water (2M of Dextrose compound). Similarly, 8.2071 grams of CaCO_3 are solvenized in a given 100 mL of deionized double distilled water to produce this CaCO_3 compound (0.82 M of CaCO_3 solution).

4.2.2 MIXING OF COMPONENTS



Fig 4.1 Dextrose solution in magnetic stirrer



Fig 4.2 Drop wise addition of CaCO_3

A Hot plate magnetic stirrer is used to combine the dextrose solution and the CaCO_3 solution, culminating in a consistent solution when the two solutions are amalgamated.

The process involves the following steps:

- Equal volumes of both CaCO_3 and dextrose solution are taken (For example, if 100 ml of glucose solution is taken, the same 100 ml of CaCO_3 solution will be taken).
- The glucose solution in vial is then put in the magnetic stirrer on the heating mantle which is shown in the fig 4.1.
- The magnetic stirred in the heated mantle is at a temperature which is set to 50°C , and the stirrer speed is set to maximum.
- Allow the dextrose solution to boil (which will approximately take 5 minutes).
- After the boiling of dextrose is seen, start adding the CaCO_3 solution taken dropwise into the beaker containing the boiling glucose solution which is shown in the fig 4.2.
- Constant mixing of both the solution will result in a gel with sticky consistency which is immediately transferred into a crucible. The image of the gel formed is shown in fig 4.3.

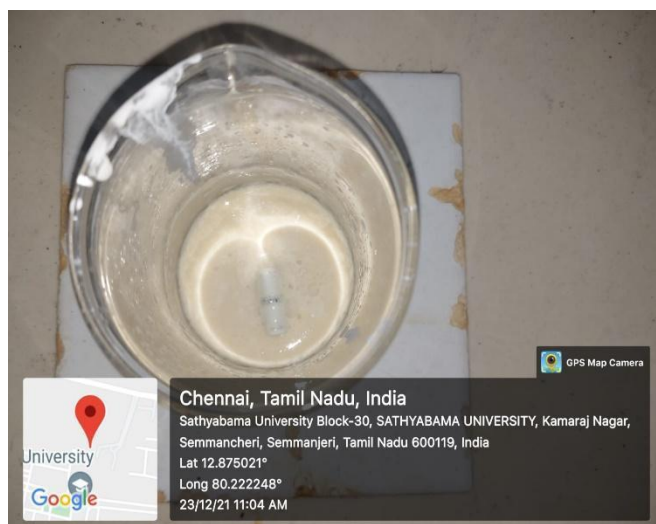


Fig 4.3 Gel formation



Fig 4.4 Gel transferred to crucible

4.2.3 REMOVAL OF MOISTURE CONTENT FROM THE GEL

The moisture content is removed from the gel formed using the Hot air oven by following the below steps:

- The crucible is placed in the oven and the temperature is set at 120° C.
- The sample in the oven is kept undisturbed for 3 hours.

The images of the gel formed containing moisture is shown on the fig 4.5 and after the removal of moisture is shown in fig 4.6 below.



Fig 4.5 Before removal of moisture

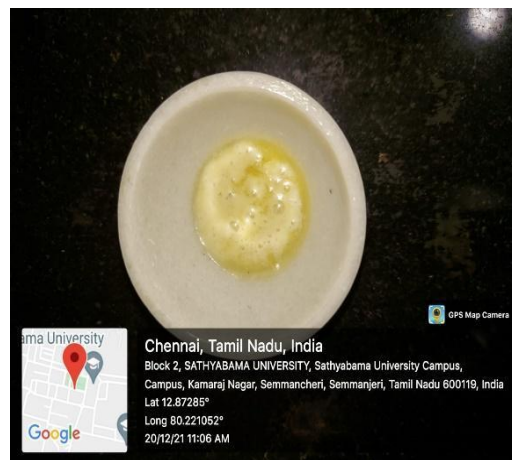


Fig 4.6 After removal of Moisture

4.2.4 PREHEATING OF SAMPLE IN A MUFFLE FURNACE

The gel free from moisture is now taken out of the oven and placed in a Muffle furnace for preheating by following the below steps:

- The crucible containing the gel is covered with an aluminium foil before placing it in the muffle furnace in order to avoid spillage that may result when subjected to high temperature in the oven.
- The covered crucible containing gel is now placed in the furnace and the temperature of the furnace is gradually increased and set at 400°C.
- Once the temperature is reached to 400°C, set the timer of the stopwatch to 7 minutes.

- Immediately after 7 minutes is reached, reduce the temperature of the furnace to room temperature 30°C.
- Switch off the furnace once the room temperature is reached.

4.2.5 SCRAPPING AND GRINDING THE SAMPLE

STEP BY STEP PROCEDURE

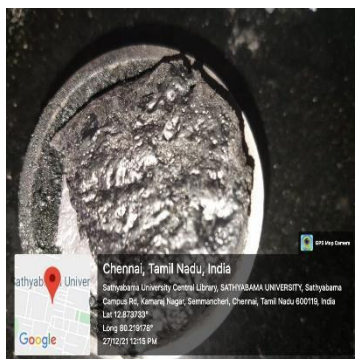


Fig 4.7 Nanocomposite before calcination



Fig 4.8 Crushed sample



Fig 4.9 Sample in mortar and pistol



Fig 4.10 Fine grinding

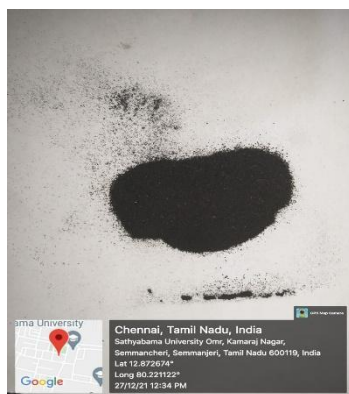


Fig 4.11 End product for calcination

The sample obtained from the muffle furnace after preheating is scrapped and grinded into a fine powder using Mortar and pistol and the end product is subjected to calcination as shown in the fig 4.11.

4.2.6 CALCINATION OF THE GRINDED SAMPLE

The grinded sample is again converted into the crucible and calcinated by following the below steps:

- In the muffle furnace, consider placing the crucible.
- The furnace's temperature is continuously grown until everything reaches 600°C.
- Once the temperature is reached to 600°C, set the timer in the stopwatch to 5 minutes
- After 5 minutes, the temperature is reduced to room temperature to 30°C
- The furnace is switched off once the room temperature is attained
- The sample is taken out of the furnace once the temperature is reduced

The image of the sample taken out of the furnace after calcination is shown in the fig 4.12.

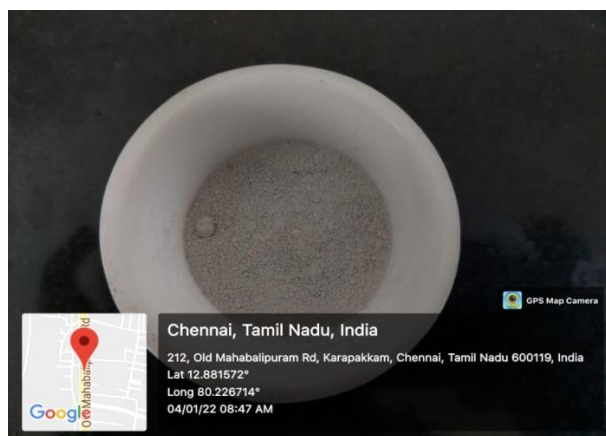


Fig 4.12 CaO/C - after calcination

The final CaO/C nanocomposite obtained is greyish in colour and stored in zip lock covers and submitted to various characterizations to study its morphology and structure followed by dye removal studies using Adsorption and isotherm studies.

4.3 CHARACTERIZATION STUDIES

4.3.1 X-RAY DIFFRACTION (XRD) ANALYSIS

The crystalline behaviour of CaO/C nanoparticles was investigated employing X-ray diffraction (XRD). In the fig 4.13, a representative X - ray diffraction pattern of CaO/C nanoparticles is shown. The crystalline structure of the produced nanocomposite is explicitly stated by the strong diffraction peaks. Peaks at $2\theta = 16.29, 17.15, 23.52, 24.73, 36.738, 42.951, 63.310, 76.441, \text{ and } 80.637^\circ$ are attributed to the CaO/C Nanocomposite lattice planes (5), (6), (2), (3), (4), (6), (9), (6), and (5). The peaks of the (6), (9), (6), and (5) lattice planes are relatively feeble in proportion to the (4) plane in the XRD patterns. The peak list of the XRD curve is given in the fig 4.14.

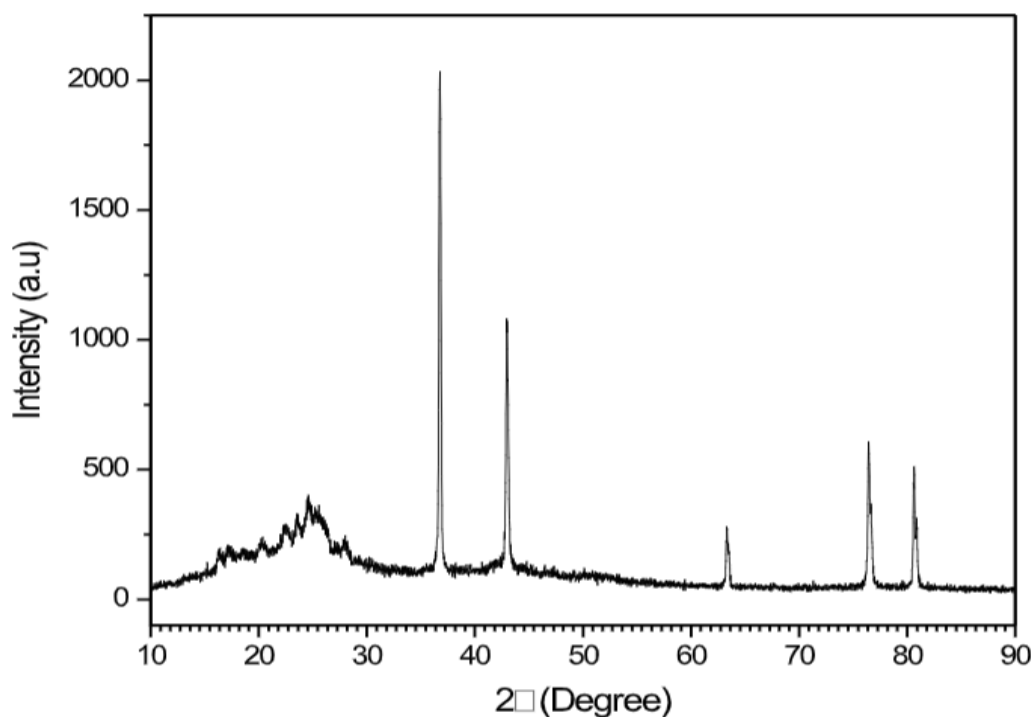


Fig 4.13 XRD Curve

Peak list

No.	2-theta(deg)	d(ang.)	Height(cps)	FWHM(deg)	Int. I(cps deg)	Int. W(deg)	Asym. factor
1	16.29(5)	5.436(16)	234(39)	0.46(9)	157(28)	0.7(2)	0.5(3)
2	17.15(6)	5.165(18)	168(33)	0.54(14)	132(25)	0.8(3)	0.5(3)
3	23.52(2)	3.780(4)	275(43)	0.21(6)	79(17)	0.29(11)	0.50(11)
4	24.73(3)	3.598(4)	624(64)	2.07(7)	1718(57)	2.8(4)	0.50(11)
5	36.738(4)	2.4443(2)	10478(264)	0.160(3)	2191(17)	0.209(7)	0.90(8)
6	42.951(6)	2.1040(3)	5333(189)	0.180(7)	1368(16)	0.256(12)	1.08(15)
7	63.310(9)	1.46779(18)	1410(97)	0.173(10)	340(9)	0.24(2)	0.76(18)
8	76.441(6)	1.24505(8)	3463(152)	0.195(6)	942(13)	0.272(16)	0.91(12)
9	80.637(5)	1.19050(6)	2973(141)	0.168(4)	682(10)	0.230(14)	1.03(14)

Fig 4.14 Peak List of XRD pattern

4.3.2 THERMAL GRAVIMETRIC ANALYSIS (TGA)

TGA characterization study is a method of determination of trace of a compound that examines the weight changes that take place when a sample is heated at a sustainable pace to evaluate a material's thermal stability and percentage of volatile component. The TGA results of the synthesized CaO/C nanocomposite is given in the fig 4.15.

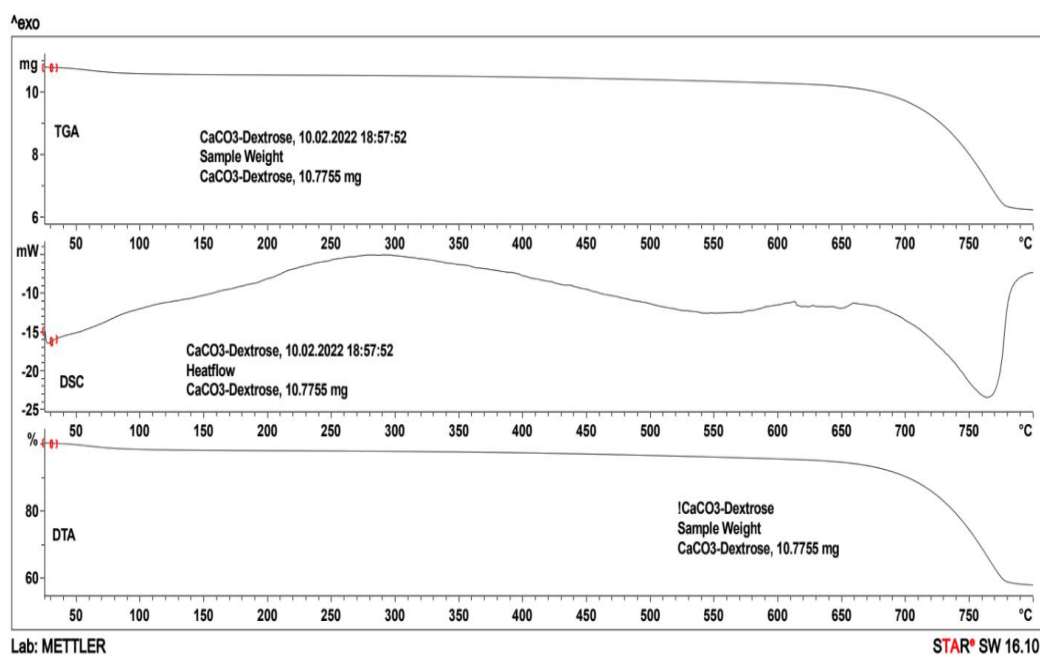


Fig 4.15 TGA Graph

During that ambient temperature, the horizontal section of the curve indicates that there is no variation in weight. And the portion of the curve where it bends and drops down signifies a shift in weight. The weight of the substance (CaCO₃-dextrose) stays constant up to 610°C, indicating that CaCO₃-dextrose is thermally stable up to that temperature. It eventually loses weight at this temperature, demonstrating that decomposition begins at this temperature. Up to 780°C, the weight of the sample decreases, but after that, the weight remains constant.

4.3.3 FOURIER - TRANSFORM INFRARED (FTIR) SPECTROSCOPY ANALYSIS

Existing functional groups incorporated in the produced CaO/C nanocomposite are investigated using the approach FTIR which is a spectroscopic analytic study. The FTIR graph results from the synthesized nanocomposite is given in the fig 4.16.

Adsorption bands were identified in various spectral regions. Additionally, at 2500 cm^{-1} , there is a little drop in the spectra, which shows the O-H stretch in the nanocomposite and the presence of atmospheric CO_2 . At 1700 cm^{-1} , a medium adsorption band is seen, indicating the C=O stretch in the nanocomposite. The C=O stretch is illustrated by a strong wide adsorption band at 1320 cm^{-1} . A significant adsorption band matching to C-H 'oop' may be detected at 850 cm^{-1} . In the FTIR graph, the Ca-O stretch occurs at 720 cm^{-1} .

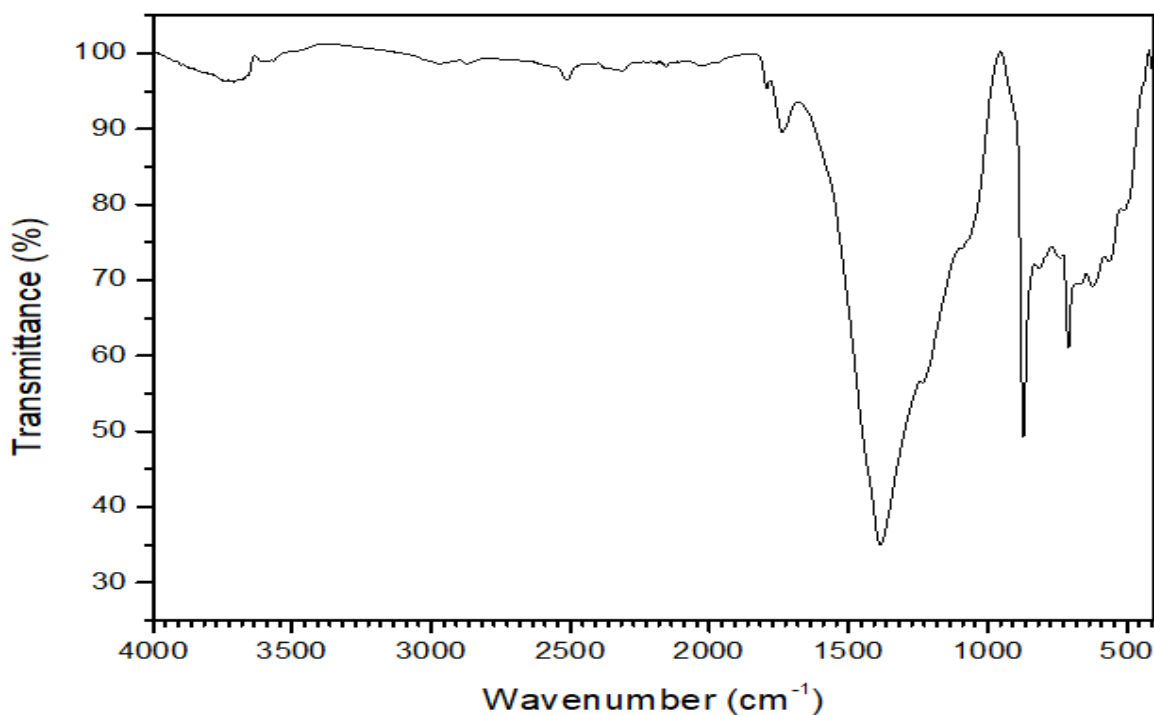


Fig 4.16 FTIR Graph

4.3.4 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

4.3.4.1 SEM OF CaO/C NANOCOMPOSITE BEFORE ADSORPTION

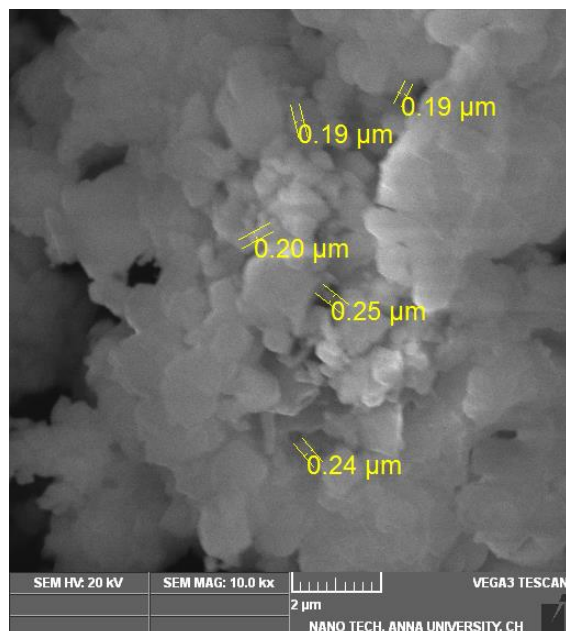


Fig 4.17 SEM Image (Before Adsorption)

The SEM characterization results of the synthesized CaO/C nanocomposite is shown in fig 4.1. The dimensions of the nanocomposites fall under the range of 190-250 nm.

4.3.5 ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDAX/EDS) ANALYSIS

4.3.5.1 EDS OF CaO/C NANOCOMPOSITE BEFORE ADSORPTION

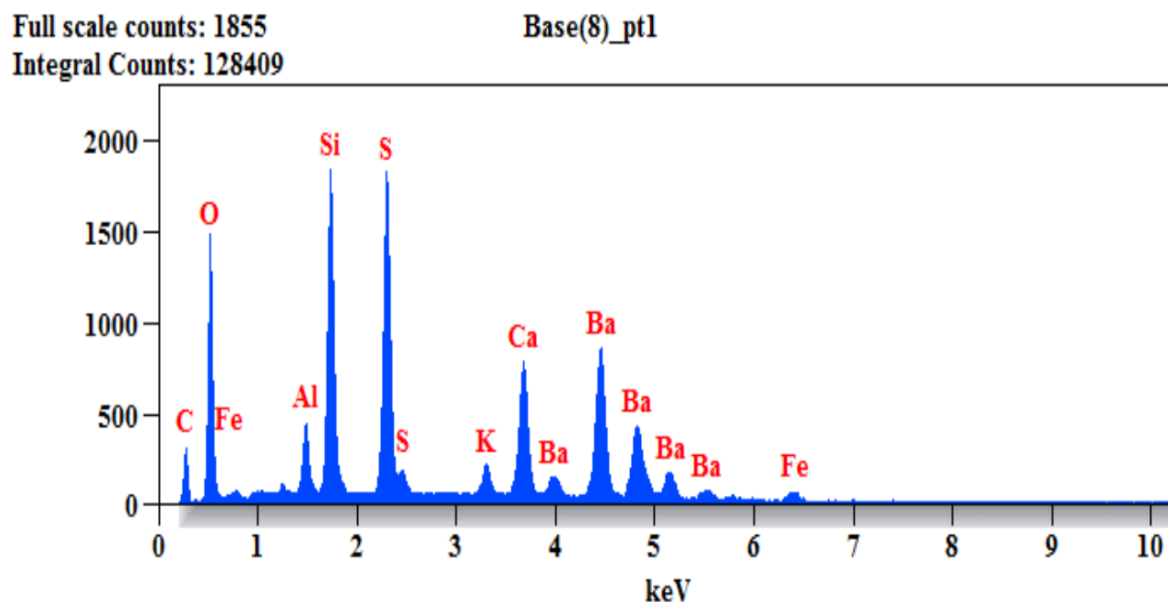


Fig 4.18 EDS Graph

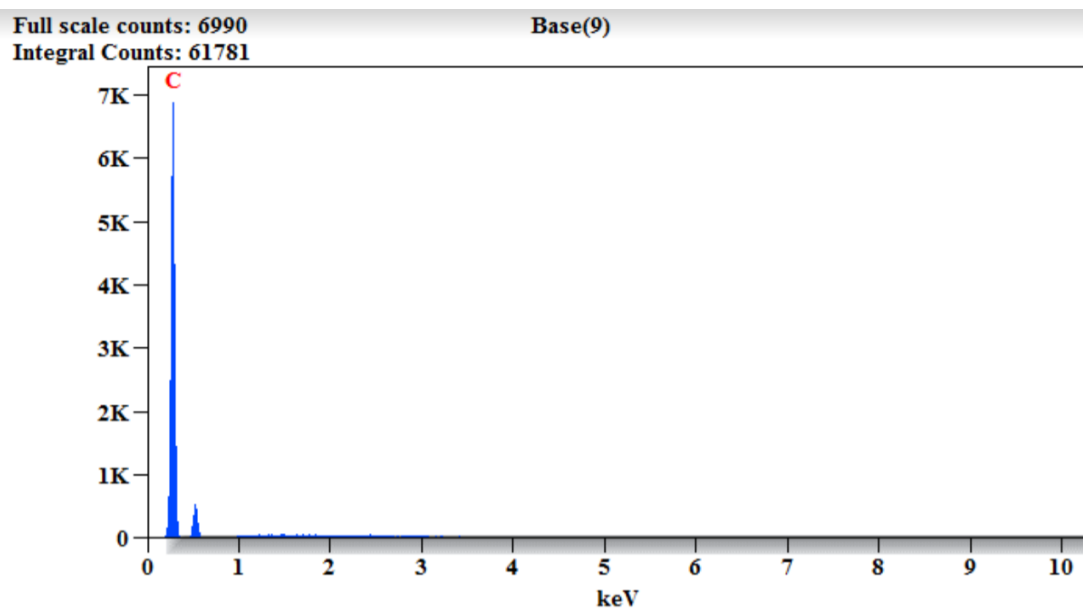


Fig 4.19 Presence of Carbon

The above fig 4.18 and 4.19 shows the EDAX patterns of the synthesized CaO/C nanocomposite and the percentage of weight of each component is given in the tabular columns. The values in the table confirms the presence of high carbon

content in the synthesized nanocomposite along with oxygen content. The high presence of carbon content in the nanocomposite is occurred as a result of high calcination temperature subjected on the sample during the synthetization process.

CHAPTER – 5

RESULTS AND DISCUSSION

The following Adsorption studies are performed on the CaO/C nanocomposite:

5.1. Effect of dosage

5.2. Effect of concentration

5.3. Effect of time

5.4. Effect of pH

5.5. Effect of temperature

The dye used for the adsorption studies was Basic orange 2 (Chrysoidine Y).

5.1 EFFECT OF DOSAGE

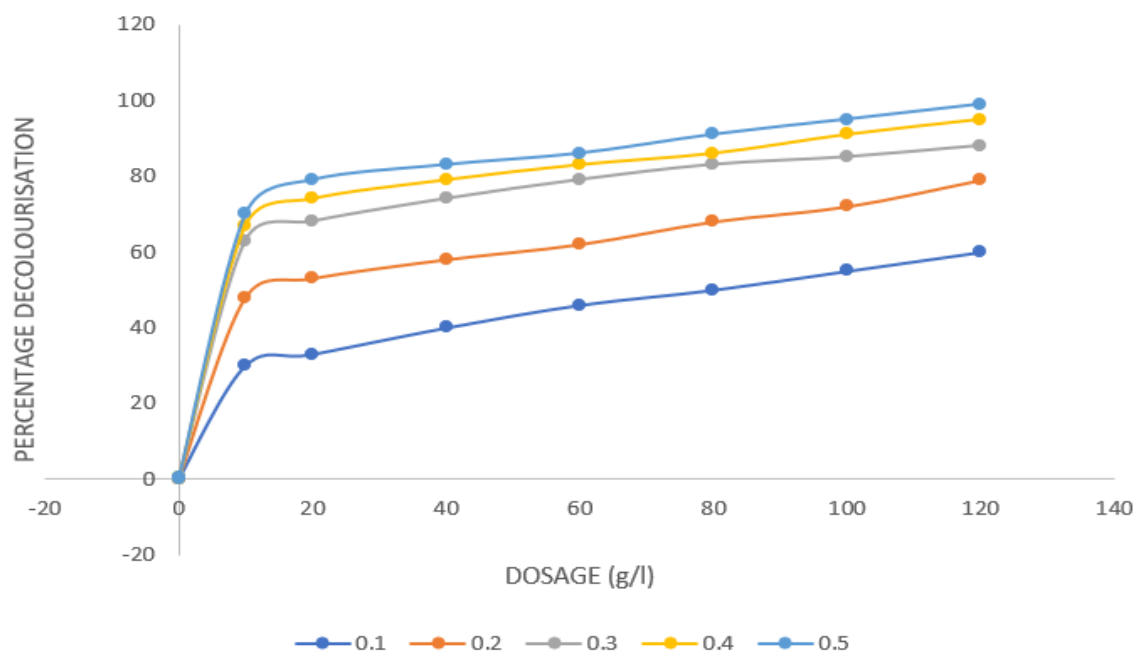


Fig 5.1 Effect of dosage

The effect of adsorbents dosage on dye removal was investigated by adding different amount of adsorbent to 100 mL of dye solution with initial dye concentration for a maximum of 120 min. The plots of the adsorption removal of dye (%) and the value of Q_e at different adsorbent dosages are shown in the graph. The acquired results revealed that dye removal efficiency elevated with increasing adsorbents doses but the inverse trend was found for the adsorption capacity (Q_e) for the synthesized nanocomposite. In fact, the increase in adsorption of CaO/C nanocomposite with adsorbent dosage can be attributed to the enhance in the adsorptive surface area and the availability of large number of active surface sites. In addition, the lower adsorption capacity at an increased adsorbent dose is due to the fact that there is the adsorption competition between adsorbent molecules and the concentration gradient get cleavage.

Existing concentration of the synthesized nanocomposite used to investigate this impact in dose on time ranged from 10 to 120 minutes. Throughout the experiment, the concentration, pH, time, and temperature were all maintained constant. The optimum dose was discovered to be 4 L/g, at which the dye adsorption was observed to be highest.

The graphical representation of the effect of dosage is shown in the above fig 5.1.

5.2 EFFECT OF CONCENTRATION

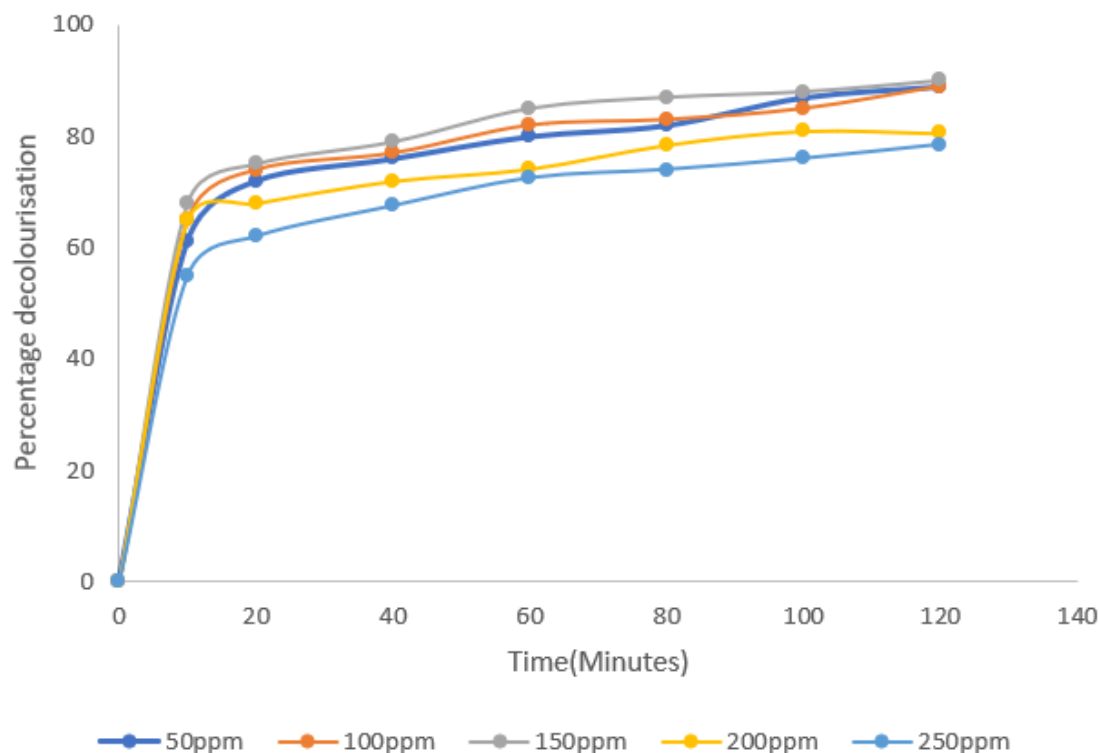


Fig 5.2 Effect of Concentration

In order to further investigate the adsorption process, the effect of initial dye concentration was studied with optimum amount of dosage, Adsorbent concentration of 50-250ppm. The relationship between the equilibrium amount of dye adsorbed per mass of adsorbents and the initial dye concentration is plotted. The results reveal that if the amount of adsorbent is remained fixed, the amount of the dye adsorbed onto nanocomposites increase with an increase in the initial dye concentration of solution. This phenomenon can be described by increasing the number of collisions between the dye ions and adsorbents, which due to the fact that the higher initial dye concentration, the higher driving force of the concentration gradient. When initial dye concentration is low, the dye molecules adsorb intensely on prepared adsorbents and the adsorption process reaches equilibrium very quickly. In other words, the ratio of initial number of dye moles to the available adsorption sites is low and subsequently the fractional adsorption becomes independent of initial concentration.

During the investigation of the efficacy of concentration, the adsorbent concentration was manipulated between 50 – 250ppm, while the dose, pH, temperature, and duration were kept constant. The optimal concentration was discovered to be 150 ppm. The graphical representation of the effect of concentration is shown in the fig 5.2.

5.3 EFFECT OF TIME

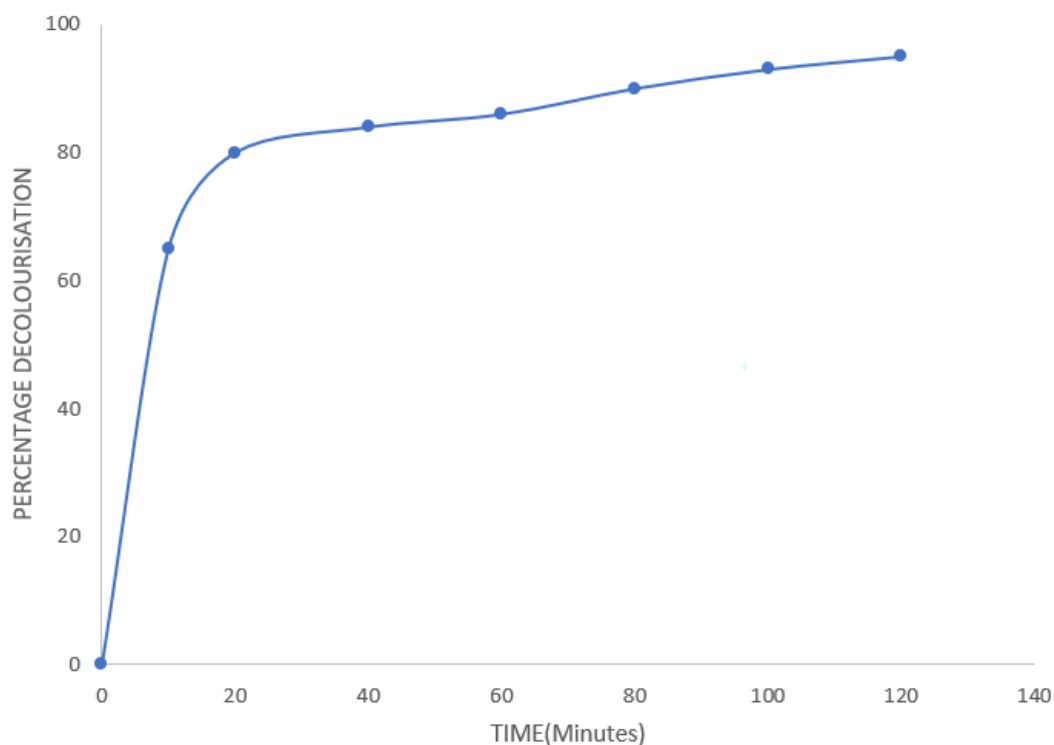


Fig 5.3 Effect of time

The time factor helps in determining the optimum time required to saturate the active adsorption sites on the surface of the nanofibers and complete removal of dye. The adsorption efficiency and adsorption capacity usually increase by increasing the contact time to reach the equilibrium state, then remains steady state thereafter. Most of the studies that was used to remove various types of dyes studied the effect of contact time and determined the time required to reach equilibrium. Therefore, the optimum contact time needed to reach the equilibrium state for the adsorption of dyes depends on the physical and chemical properties of the nanocomposites as well as the nature of the incorporated materials inside the synthesized adsorbents.

During the evaluation of concentration impact, the adsorption interval was changed from 10 to 140 minutes, while the dose, pH, temperature, and concentration were all maintained constant. The ideal time for adsorption was reported to be 60 minutes.

The graphical representation of the effect of time is shown in the fig 5.3.

5.4 EFFECT OF pH

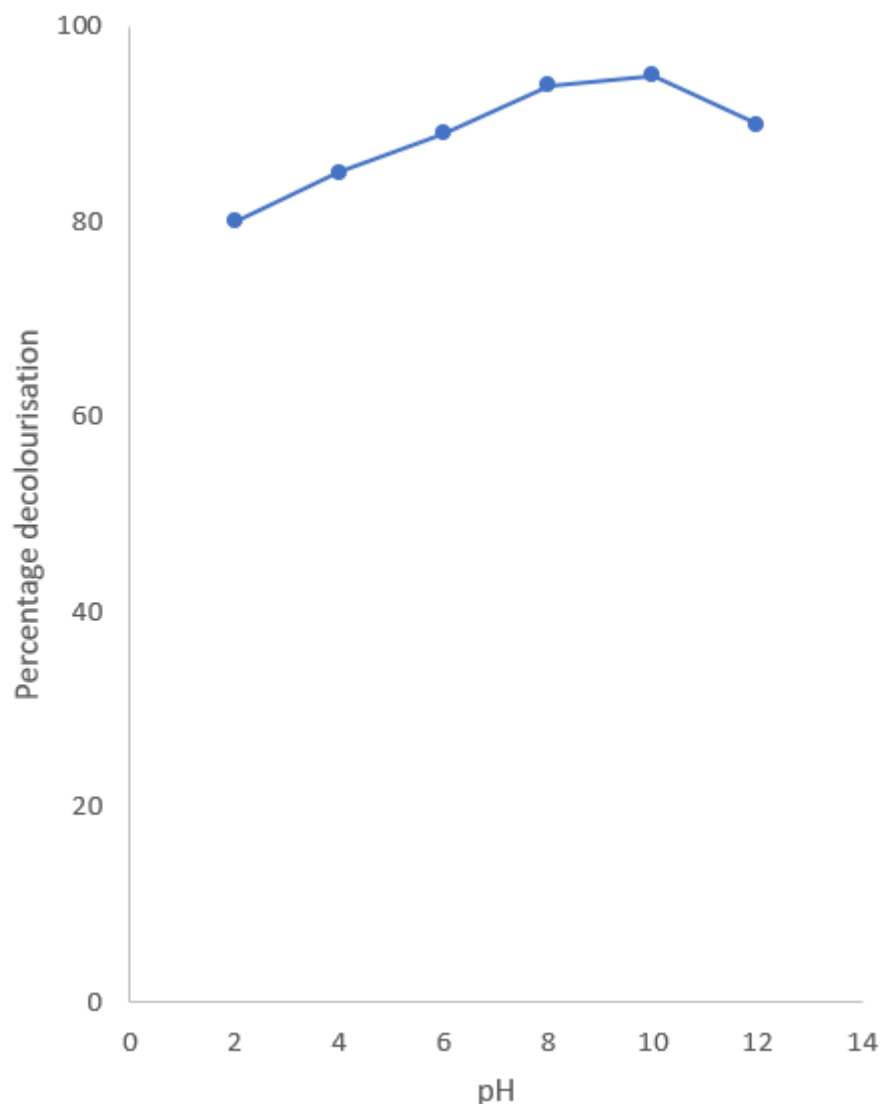


Fig 5.4 Effect of pH

The effect of initial pH was investigated in this study which can provide useful information on effects of protonation and deprotonation of prepared adsorbents on the dye removal performance. Since the main color of the selected dye changes under the alkaline condition, the experimental test of pH was varied in the range from 2 to 14 in this study. When pH was increased from 2 to 6 the equilibrium adsorption capacity remained stable without significant loss. Nonetheless, the capacity dropped sharply from at pH between 8 to 10 for the adsorbent. Such a considerable decrease could be attributed to the accumulation of proton on the

surface of adsorbents under highly basic conditions. Also, the carboxylic groups of the dye have high positive charge density at a lower pH and the number of positively charged adsorbent surface sites increased under acidic conditions. Thus, electrostatic repulsion between the positively charged adsorbent and the positively charged adsorbents surface increase with decreasing solution pH, thereby decreasing the adsorption capacity.

It was needed to ascertain the pH that correlated to the highest clearance performance. During the investigation of the impact of pH, pH of the selected pigment and adsorbent compound were differentiated from 0 to 14, while given the dose, concentration, atmosphere, and duration were maintained uniform. The ideal pH for adsorption was discovered to be between 8 and 10.

The graphical representation of the effect of time is shown in the figure 5.4.

5.4.1 POINT ZERO DISCHARGE (PZC)

The PZC survey's mission is to find the pH that corresponds to the equivalent share of two oppositely charged on the adsorbent's interface. By combining NaOH and concentrated H₂SO₄ acid in distilled water, pH solutions ranging from 2 to 14 are produced, and the appropriate quantity of amount of adsorbent is put in separate beakers holding different pH solutions. The solution is thoroughly stirred in a rotary flask shaker until being left undisturbed. The pH of the solution is measured after 24 hours, and the difference between the pH before and after 24 hours is estimated and represented as pH versus Δ pH, where Δ pH = (pH(selective variant) – pH(contaminant) after 24 hours).The below table – holds the before and after 24 hours values of pH and fig 5.5 represents the PZC graph.

Table 5.1 PZC Data sheet

Actual pH	pH after 24 hours	Δ pH
2	9.92	7.92
4	9.27	5.27
6	10.08	4.08
8	9.12	2.12
7	10.73	2.73
10	9.42	0.58
12	9.24	2.76
14	11.03	2.97

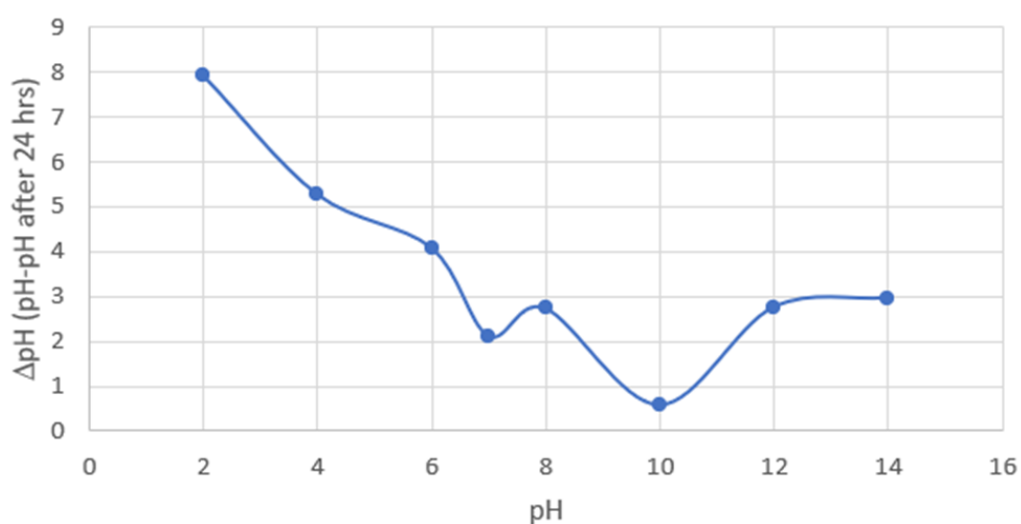


Fig 5.5 PZC Analysis

In PZC experiments, the optimum pH range for adsorption was revealed to be between 8 and 10. As a conclusion, the optimal pH range for adsorption is validated to be somewhere between 8 and 10, as determined in the Effect of pH investigation and the PZC study.

5.5 EFFECT OF TEMPERATURE

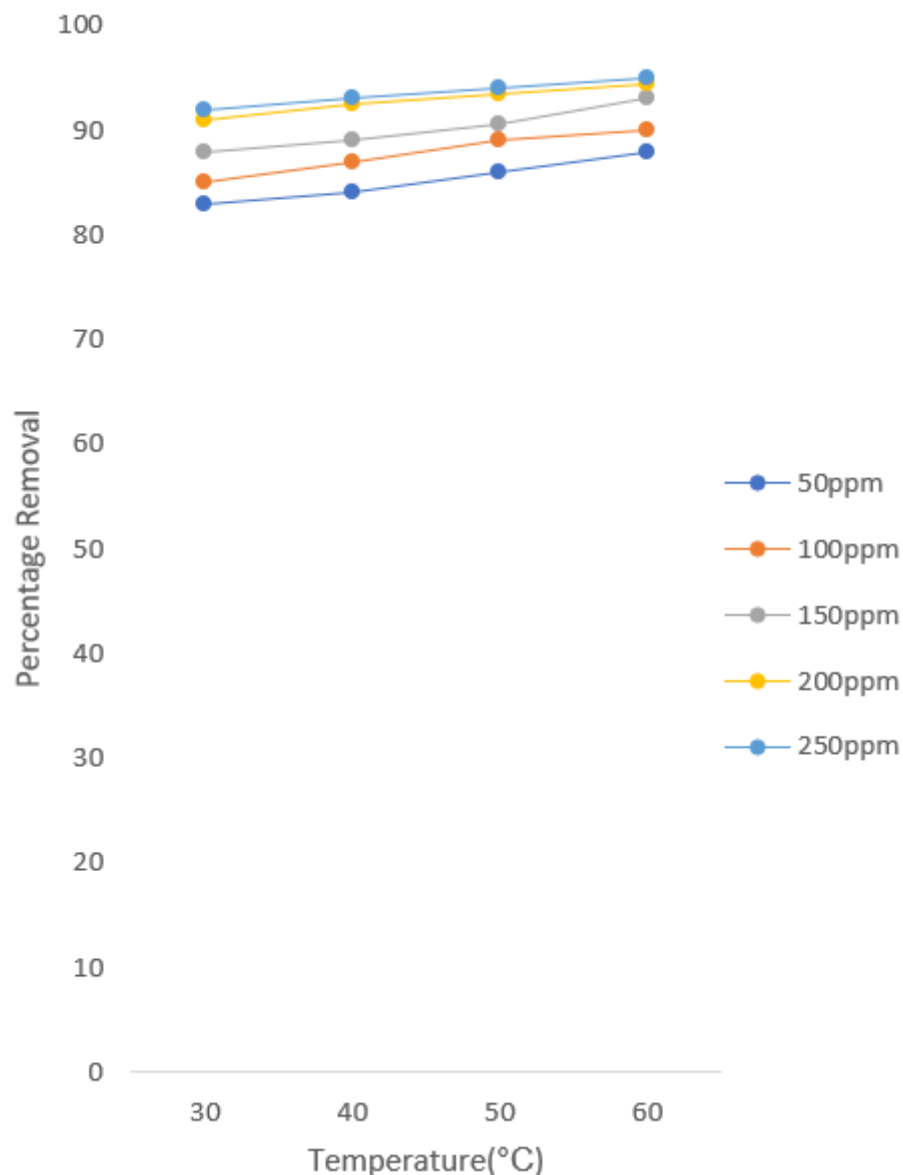


Fig 5.6 Effect of Temperature

It is obvious that the adsorption capacity increases with the increasing temperature, which indicate that the adsorption is an endothermic process. This may be related to the enhanced mobility of the dye from the bulk solution towards the adsorbent surface with increasing temperature. At higher temperature, large number of MG molecules may also obtain adequate energy to interact with active sites at the surface of adsorbents.

It was crucial to evaluate the temperature that related to the highest removal efficiency. During the investigation of the influence of temperature, the temperature of the dye and adsorbent solution was modulated from 30 to 60°C while the dose, concentration, pH, and duration were maintained constant. The optimal temperature for adsorption was discovered to be between 20 and 30 degrees Celsius. The graphical representation of the effect of temperature is shown in the fig 5.6.

5.6 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

5.6.1 SEM OF CaO/C NANOCOMPOSITE AFTER ADSORPTION

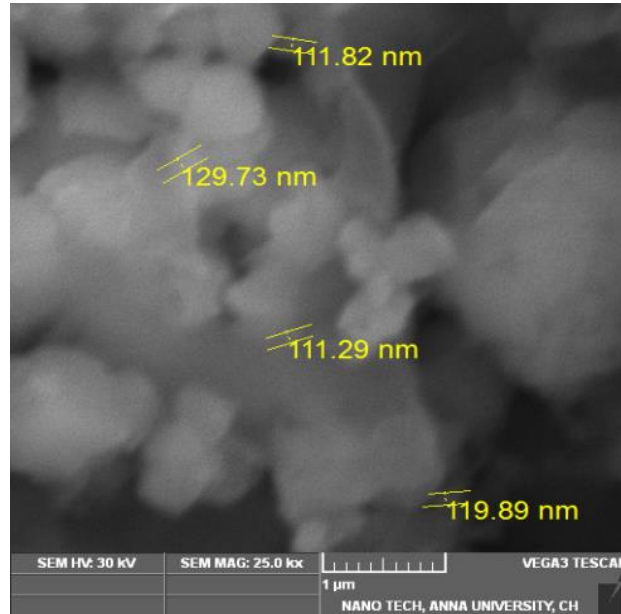


Fig 5.7 Sem image (After Adsorption)

The SEM characterization results of the synthesized CaO/C nanocomposite is shown in fig 5.7. The dimensions of the nanocomposites fall under the range of 110-130 nm.

5.7 ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDAX/EDS) ANALYSIS

5.7.1 EDS OF CaO/C NANOCOMPOSITE AFTER ADSORPTION

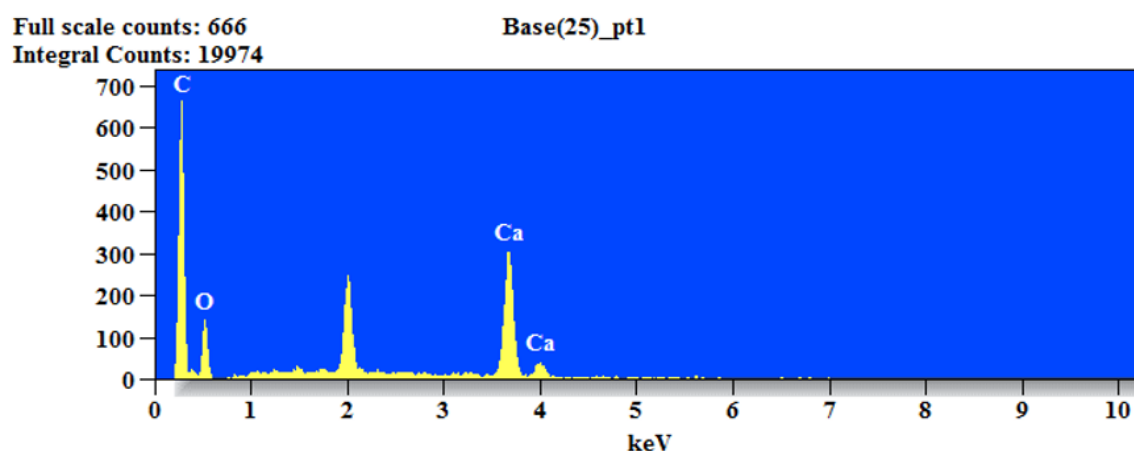


Fig 5.8 EDS After adsorption

The above fig 5.8 shows the EDAX patterns of the synthesized CaO/C nanocomposite and the percentage of weight of each component is given in the tabular columns. The values in the table confirms the presence of the Chrysoidine Y ($C_{12}H_{13}N_4Cl$). The high presence of carbon content in the nanocomposite is occurred as a result of high calcination temperature subjected on the sample during the synthetization process.

5.8 ADSORPTION ISOTHERMS

5.8.1 LANGMUIR ISOTHERM

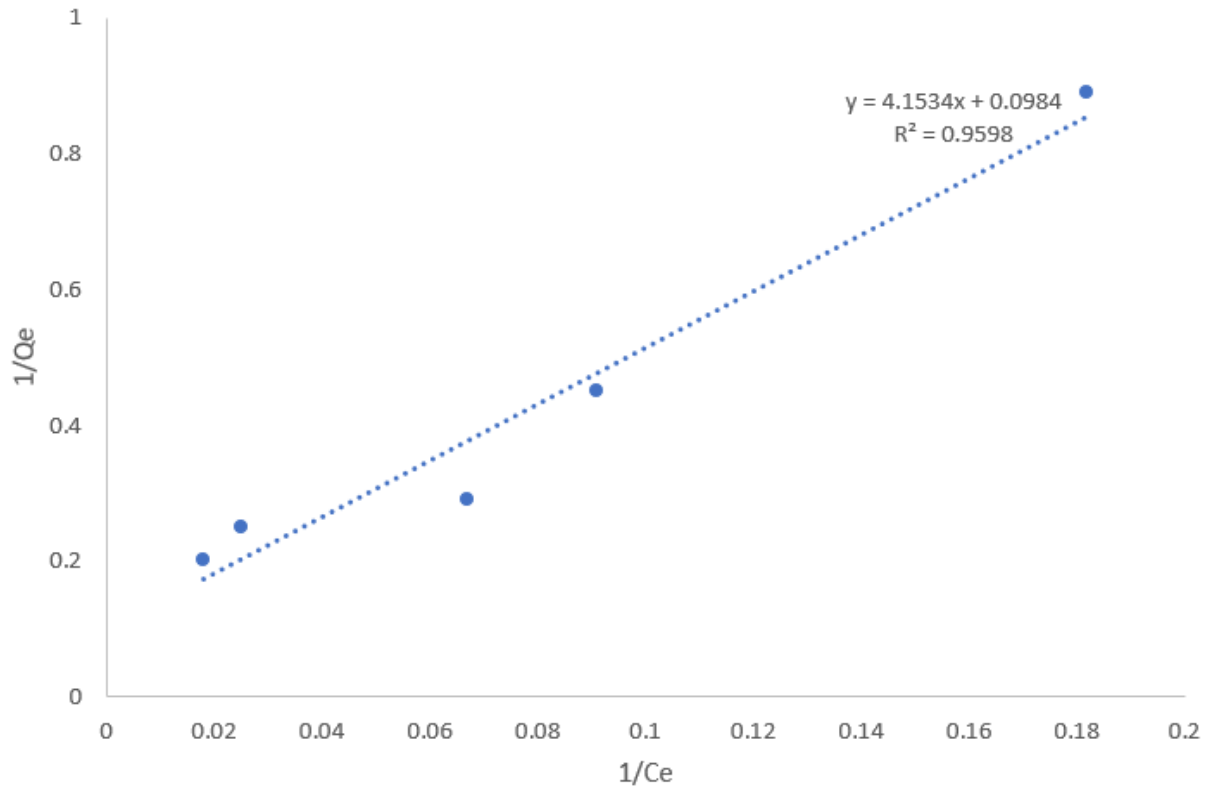


Fig 5.9 Langmuir isotherm

Table 5.2 Langmuir Isotherm

SLOPE	INTERCEPT	Q_m	K_L	R_L	R^2
4.1534	0.0984	10.1626	0.023691	0.219601	0.9598

Monolayer adsorption is characterized by the Langmuir isotherm. The equation for the Langmuir isotherm is as described in the following:

$$\frac{C_e}{Q_e} = \frac{1}{K_b \cdot A_s} + \frac{C_e}{A_s}$$

where C_e is the saturated adsorptive capacity (mg/g), Q_e is the adsorption capacity of the substance (mg/g dry adsorbent), K_b is the adsorption coefficient (L/mg), and A_s is the adsorbate solution concentration (mg/L) following adsorption equilibrium.

Fig 5.9 shows a graphical illustration of the Langmuir isotherm of adsorption of our produced nanoparticles, as well as the adsorption parameters.

5.8.2 FREUNDLICH ISOTHERM

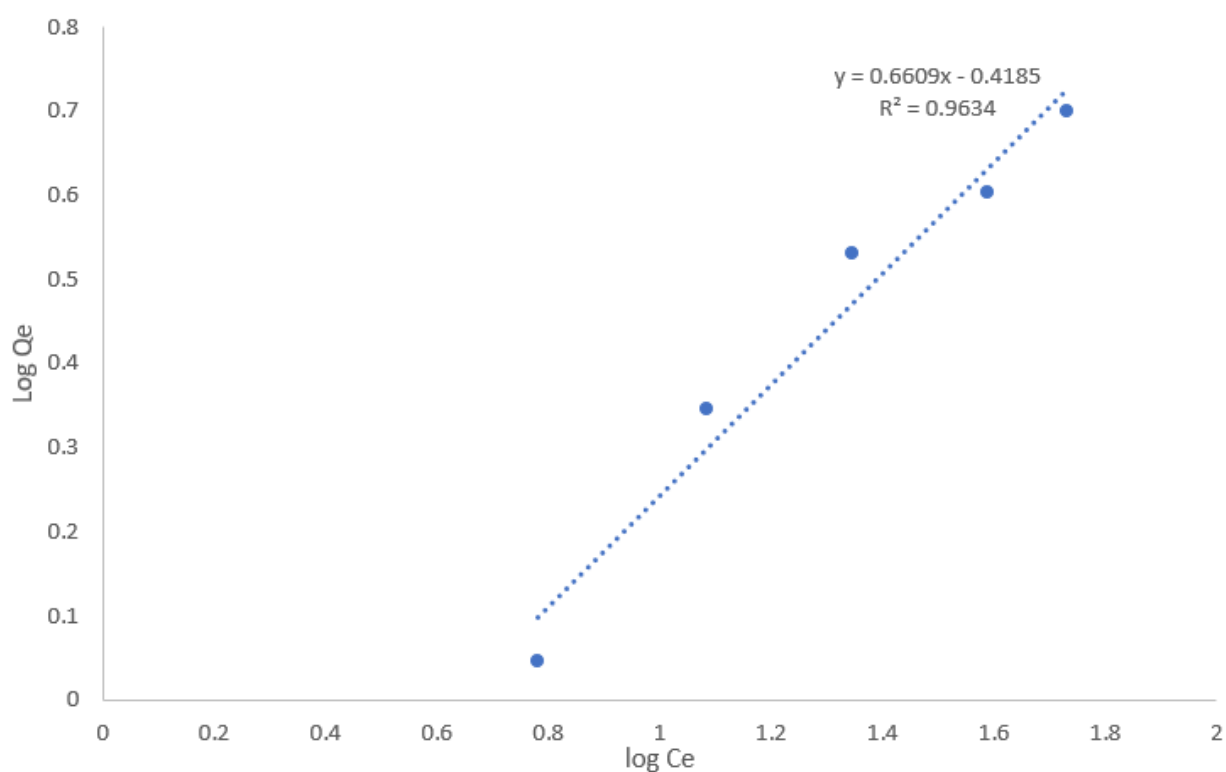


Fig 5.10 Freundlich isotherm

Table 5.3 Freundlich Isotherm

SLOPE	INTERCEPT	1/n	K _f	R ²
0.6609	-0.4185	0.6609	0.382	0.9634

Multilayer adsorption is indicated by the Freundlich isotherm. The Freundlich isotherm equation is as follows:

$$\log(Q_e) = \log K_f + \frac{1}{n} \log C$$

where K_f is the adsorption capacity, n is the adsorption intensity.

Fig 5.10 shows a graphical illustration in this Freundlich isotherm of adsorption of our generated nanoparticles, including the adsorption parameters.

5.8.3 TEMKIN ISOTHERM

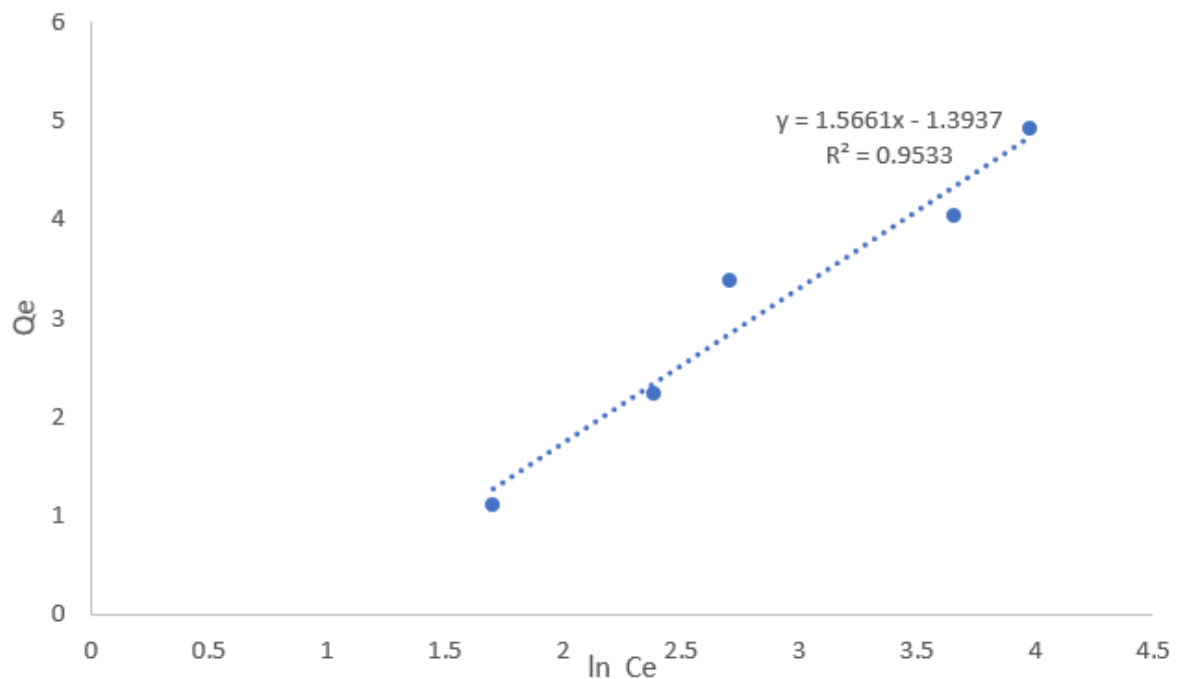


Fig 5.11 Temkin isotherm

Table 5.4 Temkin Isotherm

SLOPE	INTERCEPT	B_t	K_t	R_2
1.5661	-1.3937	1.5661	0.411	0.9533

The Temkin isotherm is given by the equation:

$$Q_e = B \ln A + B \ln C_e$$

The Temkin isotherm constant (L/g) and the heat of sorption (J/mol), correspondingly, are A and B. Such as in equation: R is the gas constant (J/mol/k),

and b is the Temkin isotherm constant associated to the energy parameter, B . e. Fig 5.11 shows a graphical illustration of the Temkin isotherm of adsorption of our generated nanoparticles, including the adsorption parameters.

5.8.4 BRUNAUER–EMMETT–TELLER(BET) SURFACE AREA ANALYSIS

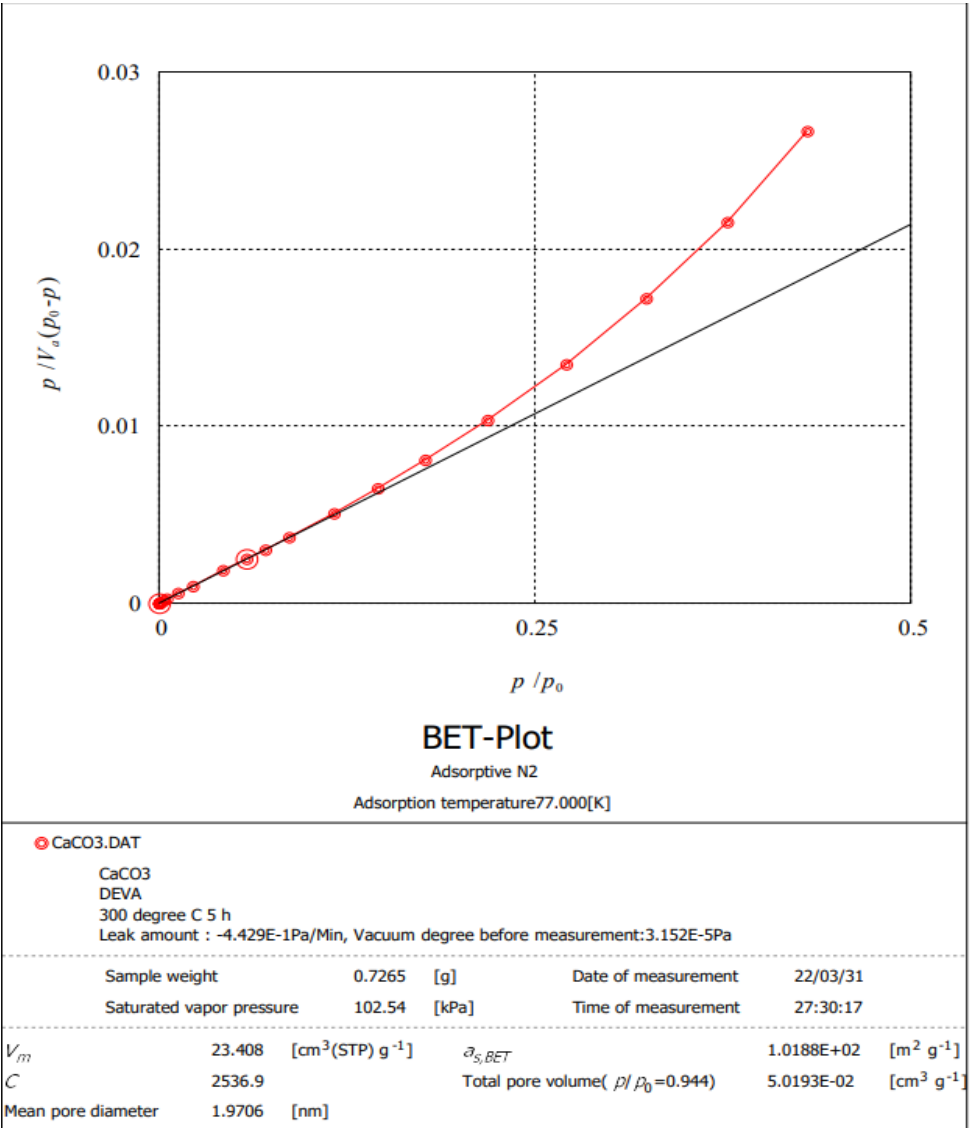


Fig 5.12 BET Isotherm Plot

The BET analysis is based on a very simple approach for an idealised adsorption process on a planar surface, where the effect of the surface affects only the first layer of adsorbed molecules, and where there are no lateral interactions within the adsorbed layer. Although it would be unusual for a material-adsorptive system to have these properties, it is found for many non-microporous materials that a fit to

the BET plot is possible in the range of P/P_0 from 0 to 0.5. However, a simple straight-line fit is insufficient to ensure validity of the model, or even consistency of the analysis. A number of recommendations have been formalised in international standards to ensure that BET analyses are reproducibly and meaningfully obtained and reported. In addition, careful sample preparation and correct use of the analysis instrument are necessary. When micro porosity is present, the fitting is normally linear in a lower pressure range, and caution is advised in the use of the term “surface area” with respect to BET results for these materials. Nanocomposites have agglomeration which cause a decrease in surface area (as BET analysis confirmed) and subsequently, decrease in dye removal percentage. The BET Surface area was detected to be $1.0188\text{E}+02 \text{ m}^2 \text{ g}^{-1}$ as given in the fig 5.12.

CHAPTER – 6

SUMMARY AND CONCLUSION

The self-propagating solution combustion process was used to make a carbon-based adsorbent – CaO/C nanocomposite – from Dextrose and Calcium carbonate. Various characterization techniques were used to investigate the structure and morphology of the produced nanocomposite. Using the SEM, the nanocomposite was discovered to be a nano-sized adsorbent. The existence of the Ca and CaO functional groups was discovered utilising FTIR analysis. The outer layer of the nanocomposite's pore found reported to be $1.0188\text{E}+02 \text{ m}^2/\text{g}$ using BET analysis. The sample's elemental composition was investigated using EDS analysis, which revealed the existence of a large quantity of carbon in the adsorbent. TGA analysis revealed that the synthesized carbon nanocomposite was thermally stable up to a temperature of 610°C .

After synthetization and characterization, adsorption tests using the Azo dye Basic orange 2 (Chrysoidine Y) were carried out to determine the best adsorption parameters.

When the concentration of the solution was 150 ppm and the dosage of the adsorbent was 4g/l for 60 minutes, the maximum adsorption of Basic orange II (Chrysoidine) from the aqueous solution using CaO/C nanocomposite adsorbent was determined to be at pH range 8-10 at temperature 20 to 30°C .

Existing isotherm models were estimated using the Langmuir, Freundlich, and Temkin isotherms, with the Freundlich isotherm having the highest R^2 value of 0.963 and the BET Surface area was detected to be $1.0188 \times 10^2 \text{ m}^2 \text{ g}^{-1}$. The mean pore diameter was found to be 1.9706 nm and the pore volume was found to be $5.0193 \times 10^{-2} \text{ cm}^3 \text{ g}^{-1}$.

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