

STUDY AND CHARACTERIZATION OF CLAY SAMPLE AVAILABLE NEAR KALPAKKAM AND ITS APPLICATION

Submitted in partial fulfillment of the requirements for the award of

Master of Science Degree in Chemistry

by

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Register No: 39910005



**DEPARTMENT OF CHEMISTRY
SCHOOL OF SCIENCE & HUMANITIES**

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of B.Fahima Begam (39910005) who carried out the project entitled **"STUDY AND CHARACTERIZATION OF CLAY SAMPLE AVAILABLE NEAR KALPAKKAM AND ITS APPLICATION"** under my supervision from December 2020 to April 2021.

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DECLARATION

I **B. FAHIMA BEGAM** hereby declare that the Project Report entitled “**STUDY AND CHARACTERIZATION OF CLAY SAMPLE AVAILABLE NEAR KALPAKKAM AND ITS APPLICATION**” done by us under the guidance of **Dr. K.CHENNAKESAVULU Ph.D.**, Associate Professor Department of Chemistry, Sathyabama Institute of Science and Technology, submitted in partial fulfilment of the requirements for the award of Master of Science degree in Chemistry.

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ABSTRACT

Soil is a complex compound of minerals, water, air, organic matter, and countless other decaying fossils. It forms on the surface of the earth. The main objective of this study was to characterize the organic constituents of Clays collected on agricultural area near Kalpakkam. The samples were collected during a cold and warm season. The collected clay sample were analyzed for their content of pH, Electrical conductivity, available phosphorus, TGA, BET surface area, SEM and FTIR measurements. The chemical composition of was determined by XRF and XRD analysis. The present study confirms the presence of excess amount of fluoride in clay, metal or metal oxides.

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LIST OF ABBREVIATION

EC	Electrical Conductivity
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
TGA	Thermo Gravimetric Analysis
BET	Brunauer- Emmett- Teller
OD	Optical Density
UV	Ultra-Violet Ray

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CHAPTER 1

INTRODUCTION

1.1 SANDY LOAM SOIL

Sandy loam soil is a type of soil used for gardening. This soil type is normally made up of sand along with varying amounts of silt and clay. Many people prefer sandy loam soil for their gardening because this type of soil normally allows for good drainage. It is important for a person not to add too much sand.

Loam is soil composed mostly of sand (particle size > 63 micrometres (0.0025 in)), silt (particle size > 2 micrometres (7.9×10^{-5} in)), and a smaller amount of clay (particle size < 2 micrometres (7.9×10^{-5} in)). By weight, its mineral composition is about 40–40–20% concentration of sand, silt, clay respectively. These proportions can vary to a degree, however, and result in different types of loam soils: sandy loam, silty loam, clay loam, sandy clay loam, silty clay loam, and loam. [1]

The only soil that is not predominantly sand, silt, or clay is called "loam". Loam soils generally contain more nutrients, moisture, and humus than sandy soils, have better drainage and infiltration of water and air than silt and clay-rich soils, and are easier to till than clay soils. The different types of loam soils each have slightly different characteristics, with some draining liquids more efficiently than others. The soil's texture, especially its ability to retain nutrients and water, are crucial.[2] Loam soil is suitable for growing most plant varieties. Bricks made of



Fig.1.1. Sandy loam soil

loam, mud, sand, and water, with an added binding material such as rice husks or straw, have been used in construction since ancient times.

1.1.1. Soil Characteristics And Texture

Soil samples were analysed for pH with a pH meter (7.57) Electrical conductivity EC (0.09ds.m⁻¹) at 25°C was determined in soil extract by using EC-meter (Hesse, 1972). Estimation of available phosphorus (73.8 ppm) was done according to the Olsen's method.

Soil texture as abiotic factor is important factors that influence distribution of minerals, organic matter retention, microbial biomass and other soil properties (Scott and Robert, 2006). Estimation of available phosphorus was done.

pH	7.57
Electrical conductivity (Ec)	26.7 mS/cm
Organic compound	0.44%
Available phosphorus	73.8ppm

Table.1.1. Characteristic and texture of my sample

1.1.2. Soil In Ancient Times

Ancient agricultural systems and soils are long-term sources of data on management strategies and land resource change. The record of early agriculture provides a deep-time perspective on land use and human environmental interaction, responses to climate change, and anthropogenic soil processes. Many past and contemporary ancient cultures have farmed the same land on scales of centuries to millennia. [2]

1.2. CLASSIFICATION OF LOAM

Ancient systems and agriculture are long-term sources of data on land management strategies and transformation. The earliest agricultural record provides an in-depth look at land use and human interactions with ecosystems, responses to climate change, and anthropogenic soil systems. Many ancient and

ancient civilizations have dominated the same world over the centuries to thousands of years.

1.3. APPLICATIONS OF LOAM

Fertile soil can be divided into certain subtypes. Other examples are sandy loam, silt loam, loamy loam, and silty clay loam. The different soil types have a variety of factors such as rock erosion and erosion that are too small to affect indigenous growth but can be important for crop growth. [3]

1.3.1. In Farming

Loam is considered ideal for gardening and agricultural uses because it retains nutrients well and retains water while still allowing excess water to drain away [4]. A soil dominated by one or two of the three particle size groups can behave like loam if it has a strong granular structure, promoted by a high content of organic matter. However, a soil that meets the textural definition of loam can lose its characteristic desirable qualities when it is compacted, depleted of organic matter, or has clay dispersed throughout its fine-earth fraction.

Pea can be cultivated in sandy loam and clay loam soils, but not more compacted sandy soils [5].

Loam is found in a majority of successful farms in regions around the world known for their fertile land. Loam soil feels soft and crumbly and is easy to work over a wide range of moisture conditions.

1.4 BACTERIAL STUDY

Antibacterial Property

There is some concern that antibiotic residues in applied manure may promote the emergence of antimicrobial organisms in the environment. Soil-marketed antibiotics have been found to maintain their antimicrobial properties because both antibiotics inhibited the growth of all bacterial species.

The main characteristics of the abiotic are climate, including temperature, humidity, soil texture, soil composition, salt and pH. Climatic conditions also affect the body of soil organisms as they vary around the world and, in the same areas, during the seasons.

CHAPTER 2

LITERATURE SURVEY

1.Title: Soil Thermal Properties at Kalpakkam in Coastal India.

Author : K. Ananda Kumar, R. Venkatesan, Thara V. Prabha

Journal and Year : *Journal of earth system and science* ,2001

Inference: Time series of soil surface and subsurface temperatures, soil heat flux, net radiation, air temperature and wind speed were measured at two locations in Kalpakkam, coastal southeast India. The data were analysed to estimate soil thermal diffusivity, thermal conductivity, volumetric heat capacity and soil heat flux.

2.Title: Qualitative and quantitative characterization of Brazilian natural and organophilic clays by thermal analysis.

Author : J. Dweck, Bloco E Sala

Journal and Year : *Journal of thermal analysis and calorimetry*, 2008

Inference: Differential thermal analysis (DTA) was the first thermal analysis technique, which was used to characterize and to qualitatively distinguish natural clays with a same chemical composition but with different molecular structures.

3.Title: Analytical characterization of natural clay.

Author: Refat El- Sheiky, Moslech Al- Shamrani.

Journal and Year : *American institute of physics*,2010

Inference: The current paper introduces the study of morphology and electronic microscopy characterization of one type of the smectite Saudi nano clay montmorillonite type. During the last decade, nanotechnology achieved a recognized progress in many fields based mainly on synthesized materials. Much attention is devoted to produce natural nanoparticles. It was found that the clay is one of the rare materials which have platelets of nano scale size. The nano clay minerals are found in different types. It is investigated that the nano clay minerals have super properties which cannot be found in the other materials. The Kingdom of Saudi Arabia has many zones having different types of good nano clays. These nano clays are found in certain mixtures with other different materials such as Mg, Ca, Fe and others. By developing an innovated technique, we could extract Saudi

Arabian nano clay with high grade purity. The results are very interesting. The produced nano clay particles are with good quality and super properties. It can be used in many fields of nanocomposites.

4. Title : Geo chemical and thermodynamic aspects of sorption of Strontium on Kaolinite dominated clay samples at Kalpakkam.

Authors : R. Deepthi Rani, P. Sasidhar

Journal and Year: *Environmental earth sciences*, 2011

Inference : The mobility of strontium in subsurface is largely influenced by sorption on to clay minerals. In the present study, kaolinite clay samples collected from the Kalpakkam nuclear plant site were employed to understand the sorption characteristics of strontium by batch method. The effect of several parameters such as time, strontium ion concentration, pH, temperature and ionic strength was investigated.

5. Title : Instrumental characterization of clay by XRF, XRD and FTIR

Authors: Preeti sagar Nayak and B K Singh

Journal and Year: *Bulletin of materials science*, 2007

Inference: Natural clay minerals are well known and familiar to mankind from the earliest days of civilization. Because of their low cost, abundance in most continents of the world, high sorption properties and potential for ion exchange, clay materials are strong candidates as adsorbents. Clay is composed mainly of silica, alumina and water, frequently with appreciable quantities of iron, alkalies and alkali earths (Ralph 1968). Two structural units are involved in the atomic lattices of most clay minerals. One unit consists of closely packed oxygens and hydroxyls in which aluminium, iron and magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygens or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a hexagonal network that is repeated indefinitely to form a sheet of composition, $\text{Si}_4\text{O}_6(\text{OH})_4$.

6. Title: Effects of soil texture on chemical compositions, microbial population and carbon mineralization in soil.

Authors: Najmadeen H. Hamarashid, Mohammad A. Othman, Mohamed-Amin H. Hussain

Journal and Year: *Egyptian journal of experimental biology*, 2010

Inference: Soil texture plays a key role in carbon storage and strongly influences nutrient retention and availability. The objective of this study was to 1) determine the effects of soil texture on soil chemical compositions and abundance of microbial communities in soils collected from nine different localities of Sulaimani governorate, 2) The correlation between mineralization of carbon and tested parameters.

7. Title : Anthropogenic Soil Change in Ancient and Traditional Agricultural Fields in Arid to Semiarid Regions of the America

Authors: Sandor, Jonathan A., and Homburg, Jeffrey A.

Journal and Year: *Society of Ethnobiology*, 2017

Inference: Soils form the foundation for agriculture and are changed by farming through active management and unintentionally. Soil change from agriculture ranges from wholesale transformation to ephemeral and subtle modification. The archaeological record of early agricultural systems holds information about soil change on centurial to millennial scales, with important implications for long-term soil condition and land use sustainability. Knowledge of early agricultural management can also be inferred from soils, including farming strategies in dynamic, challenging environments.

8. Title: How to Read and Interpret FTIR Spectroscopy of Organic Material

Authors: Asep Bayu Dani Nandiyanto*, Rosi Oktiani, Risti Ragadhita

Journal and Year: *Indonesian Journal of Science & Technology*, 2019

Inference: Fourier Transform Infrared (FTIR) has been developed as a tool for the simultaneous determination of organic components, including chemical bond, as well as organic content. The step-by-step method on how to read the FTIR data was also presented, including reviewing simple to the complex organic materials. This study is potential to be used as a standard information on how to read FTIR peaks in the biochemical and organic materials.

CHAPTER 3

AIM AND SCOPE

The clay sample near kalpakkam area were collected and characterized for the following

- Chemical entities present in the clay by FTIR
- Morphology by SEM
- Surface area of the clay by BET measurements
- Thermal stability by TGA
- The presence of minerals was further tested by XRD studies
- To know the chemical composition of mineral present in the clay by using XRF
- Antibacterial property of clay using OPTICAL DENSITY Measurement
- Amount of fluoride detection in water and ragi.

CHAPTER 4

MATERIALS AND METHOD

4.1.MATERIALS

4.1.1. SOIL SAMPLING

The sample have been collected at the month of November, sample is collected nearby village of kalpakkam the sediment sample collected at five wet areas, each weighed about 1 kilogram. The sample was also collected from a depth of 4-5 cm from the surface, after removing naked stones, plant and animal debris, these composite sample were dried and sieved through a mesh. Before analysis samples were placed in plastic pouches. Then soil was subjected to the chemical, physical and biological analysis. The location map is given below.

4.2 CHARACTERIZATION TECHNIQUE

The chemical, physical and biological characteristics of the clay are essential to identify the specific properties presence.

4.2.1. FOURIER TRANSFORM INFRARED SPECTROSCOPY - (FTIR)

FTIR is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high-resolution spectral data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time. Infrared spectroscopy exploits the fact that molecules absorb frequencies that are characteristic of their structure. These absorptions occur at resonant frequencies, i.e., the frequency of the absorbed radiation matches the vibrational frequency. The energies are affected by the shape of the molecular potential energy surfaces, the masses of the atoms, and the associated vibronic coupling. In particular, in the Born–Oppenheimer and harmonic approximations, i.e., when the molecular Hamiltonian corresponding to the electronic ground state can be approximated by a harmonic oscillator in the neighbourhood of the equilibrium molecular geometry, the resonant frequencies are associated with the normal modes corresponding to the molecular electronic ground state potential energy surface.

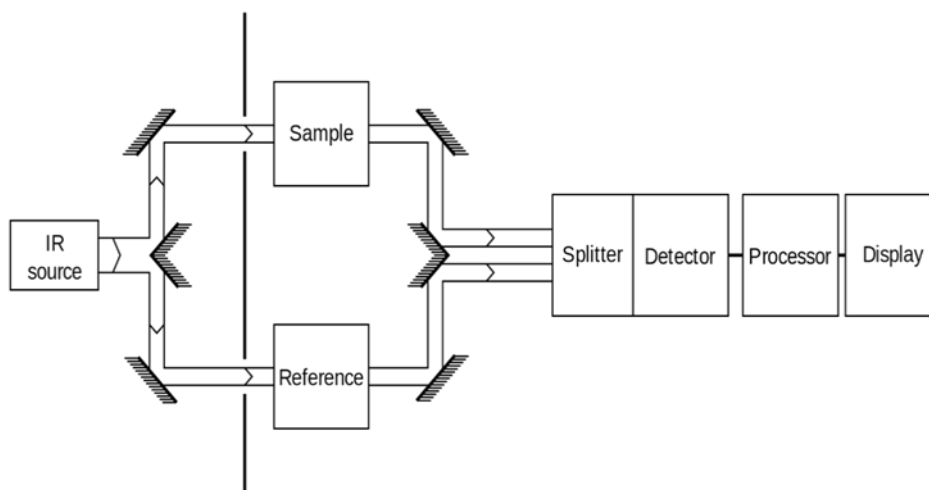


Fig.4.1. FTIR absorption spectrometer

In short, the IR spectrum is divided into three wavenumber regions: far-IR spectrum ($<400\text{ cm}^{-1}$), mid-IR spectrum ($400\text{--}4000\text{ cm}^{-1}$), and near-IR spectrum ($4000\text{--}13000\text{ cm}^{-1}$). The mid-IR spectrum is the most widely used in the sample analysis, but far- and near-IR spectrum also contribute in providing information about the samples analysed. This study focused on the analysis of FTIR in the mid-IR spectrum. The mid-IR spectrum is divided into four regions: (i) the single bond region ($2500\text{--}4000\text{ cm}^{-1}$), (ii) the triple bond region ($2000\text{--}2500\text{ cm}^{-1}$), (iii) the double bond region ($1500\text{--}2000\text{ cm}^{-1}$), and (iv) The fingerprint region ($600\text{--}1500\text{ cm}^{-1}$).

Identifying the fingerprint region ($600\text{--}1500\text{ cm}^{-1}$) This area is typically specific and unique. See detailed information in Table 1. But several identifications can be found: (1) Between 1000 and 880 cm^{-1} for multiple band absorption, there are absorption bands at 1650 , 3010 , and 3040 cm^{-1} . (2) For C-H (out-of-plane bending), it should be combined with absorption bands at 1650 , 3010 , and 3040 cm^{-1} which show characteristics of compound unsaturation. (3) Regarding vinyl-related compound, about 900 and 990 cm^{-1} for identifying vinyl terminals (--CH=CH_2), between 965 and 960 cm^{-1} for trans unsaturated vinyl (CH=CH), and about 890 cm^{-1} for double olefinic bonds in single vinyl (C=CH_2). (4) Regarding aromatic compound, a single and strong absorption band is around 750 cm^{-1} for ortho and 830 cm^{-1} . [10]

4.2.2. SCANNING ELECTRON MICROSCOPE - (SEM)

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. Specimens are observed in high vacuum in a conventional SEM, or in low vacuum or wet conditions in a variable pressure or environmental SEM, and at a wide range of cryogenic or elevated temperatures with specialized instruments.

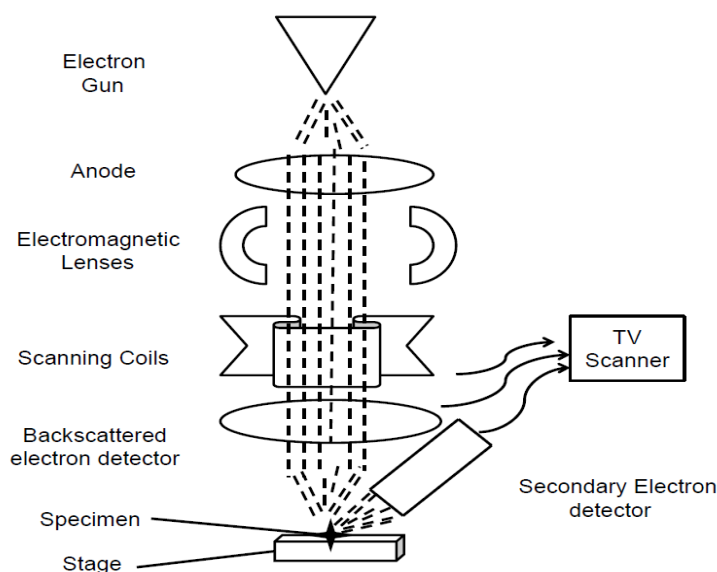


Fig.4.2.Scanning Electron Microscope

4.2.3. THERMO GRAVIMETRIC ANALYSIS – (TGA)

Thermo Gravimetric Analysis or Thermal Gravimetric Analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption, adsorption and desorption; as well as chemical phenomena including chemisorption's, thermal decomposition, and solid-gas reactions (e.g., oxidation or reduction)

The upper temperature used for TGA is normally 1000 °C. Quantitative analysis is done from the thermal curve by measuring the loss in mass m.[12]



Fig 4.3. Thermo Gravimetric Analyzer

4.2.4. X-RAY DIFFRACTION ANALYSIS – (XRD)

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. It is used to study the structure, composition, and physical properties of materials. XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that leave the material (X-ray diffractometer).

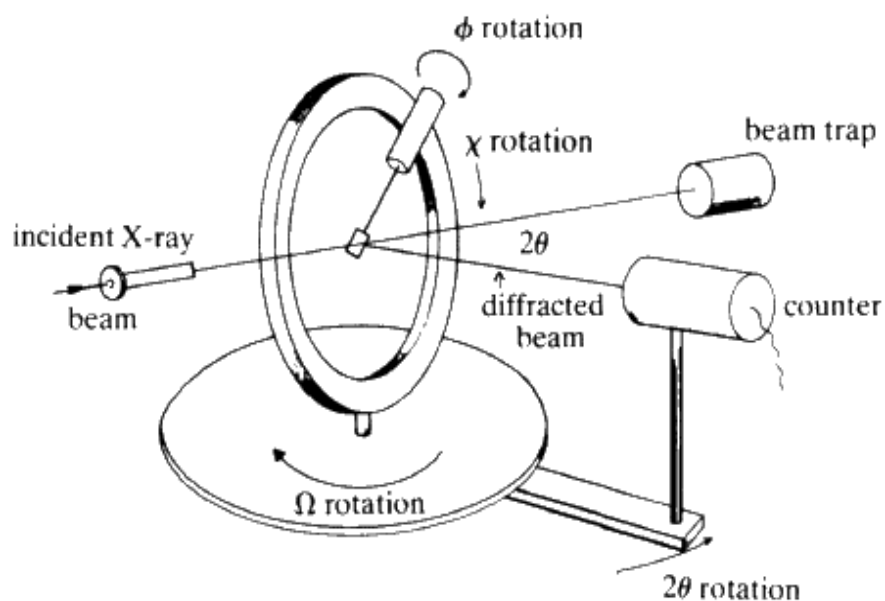


Fig 4.4. X-ray diffractometer

4.2.5. POROSIMETRY

Porosimetry is an analytical technique used to determine various quantifiable aspects of a material's porous structure, such as pore diameter, total pore volume, surface area, and bulk and absolute densities. Brunauer-Emmett-Teller (BET) theory aims to explain the physical adsorption of gas molecules on a solid surface and serves as the basis for an important analysis technique for the measurement of the specific surface area of materials.



Fig.4.5.Surface area and pore size analyser

BET method actually means the analysis of isotherm data by a method developed by Brunauer, Emmett and teller. by means of the BET equation the amount of adsorbed gas, which build up one monolayer on the surface, can be calculated from the measured isotherm. The number of molecules in this monolayer multiplied by the required space of one molecule gives the BET surface area. Besides the adsorption of nitrogen at 77 k, krypton adsorption at 77k is recommended for the determination of very small surface area.

- The choice of the measuring instrument depends on: Scope of the task (only for BET) surface area or also the pore volume and pore size distribution) [13]
- The range of the surface area of the materials (the determination of very small surface areas y krypton adsorption requires a special measuring technique (Chennakesavulu et al. 2010).[13]

- The desired throughput (1,2,3 or 4 station instrument).

4.2.6 X-RAY FLUORESCENCE – (XRF)

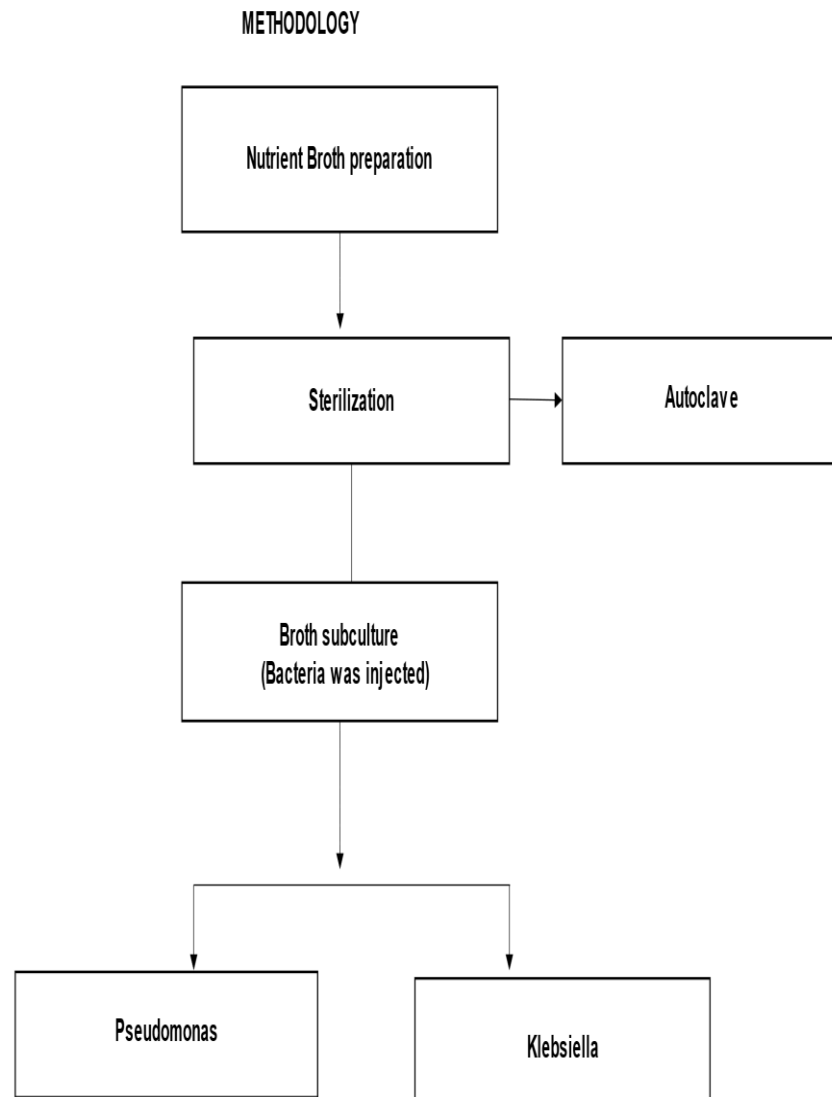
X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by being bombarded with high-energy X-rays or gamma rays. The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science, archaeology and art objects such as paintings.

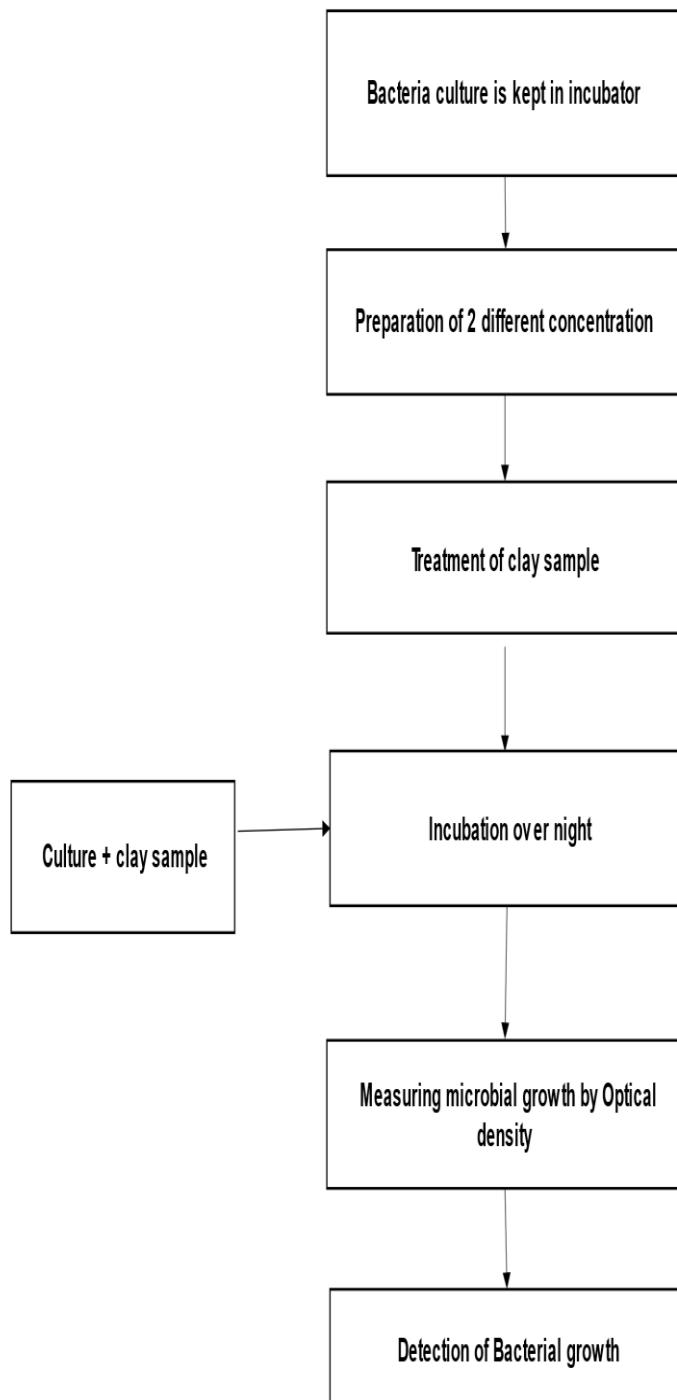
XRF (and particularly EDXRF) is ideally suited for very fast qualitative elemental analysis. Typically, all elements from sodium through to uranium can be detected simultaneously, with good quality spectra obtained in seconds/minutes.

The X-ray fluorescence process

1. A solid or a liquid sample is irradiated with high energy X-rays from a controlled X-ray tube.
2. When an atom in the sample is struck with an X-ray of sufficient energy (greater than the atom's K or L shell binding energy), an electron from one of the atom's inner orbital shells is dislodged.
3. The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher energy orbital shells.
4. The electron drops to the lower energy state by releasing a fluorescent X-ray. The energy of this X-ray is equal to the specific difference in energy between two quantum states of the electron. The measurement of this energy is the basis of XRF analysis [17]

4.3.METHODOLOGY





4.3.1 Broth Subculture Method

Sub-culture is a procedure of transferring of microorganism into fresh nutritive medium from its stock culture. It includes transfer of culture from slant to slant, slant to plate, plate to plate, plate to slant, solid medium to broth, and broth to solid media. sub-culturing is done to maintain culture in its active form (prolonging life and/or increase the number of cells) for varied applications. Broth cultures are liquid cultures used to grow bacteria in laboratories. To create a broth culture, a scientist begins with a sterile liquid growth medium. The medium is inoculated with bacteria and placed in an incubator at the appropriate temperature.

4.3.2 Preparation of Nutrient Broth

Add 1.95g of nutrient broth powder in 150ml of distilled water. Mix and dissolve them completely in a conical flask, then pipped out 30 ml in each 6 sterile glass culture tubes and labelled (p_{con}, p_{0.5}, p_{1.0}, k_{con}, k_{0.5}, k_{1.0}). Sterilize by autoclaving at 121°C for 15 minutes.

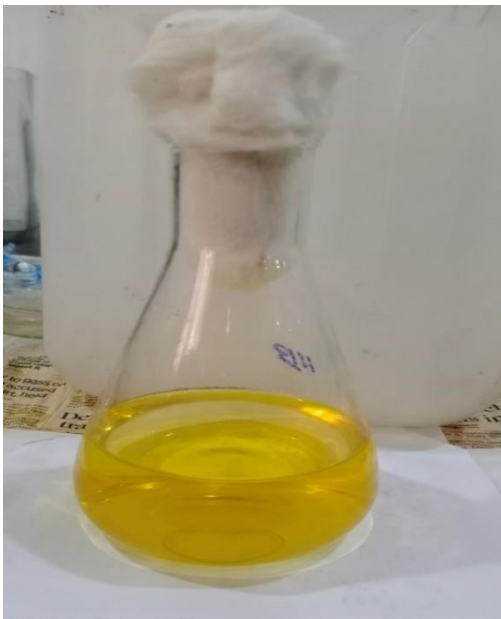


Fig.4.6.a. Nutrient broth



Fig.4.6.b. 30ml pipped Nutrient broth

4.3.3 Sterilization Of Nutrient Broth

Bottled media for the sterility test requires sterilization after preparation. Bottled media is typically sterilized by autoclaving. with the sterilization of bottled media, this is typically carried out in a steam autoclave at temperatures between 121-134°C. However, the cycle needs to be developed with not only sterilization in mind but also to avoid damage to the medium through the heating process. The heat-treatment of culture media, which contain peptides, sugars, minerals and metals, can result in nutrient destruction if the heat is too great. After the heating process we have to cool down the sterilized media, the cool-down time is dependent on the size of the load in the chamber and the heat loss rate from the autoclave.

4.3.4 Incubation

An incubator is a device used to grow and maintain microbiological cultures or cell cultures. The incubator maintains optimal temperature, humidity and other conditions such as the CO₂ and oxygen content of the atmosphere inside. Incubators are essential for much experimental work in cell biology, microbiology and molecular biology and are used to culture both bacterial and eukaryotic cells. The cooled sterilized tubes should be incubating for an overnight of 39 degree Celsius.

4.3.5 Bacterial Inoculation

Pick up a tube of sterile nutrient broth with your free hand, carefully remove the cap (cotton plug) with your little finger of the hand holding the loop, Heat the inoculating loop in the spirit lamp until it is red hot. Heat several inches of the loop, since that much of it will contact the inside of the tubes. Allow the loop to cool for a few seconds while you hold it in your hand. Do not put it down or allow the loop to touch any surface after it is sterile and flame (spirit lamp) the mouth of the tube. Insert the loop into the sterile broth and inoculate it by gently moving the loop back and forth in order to disperse the cells. This process should be done in the air flow chamber to avoid the contamination of microbes that surrounds us.

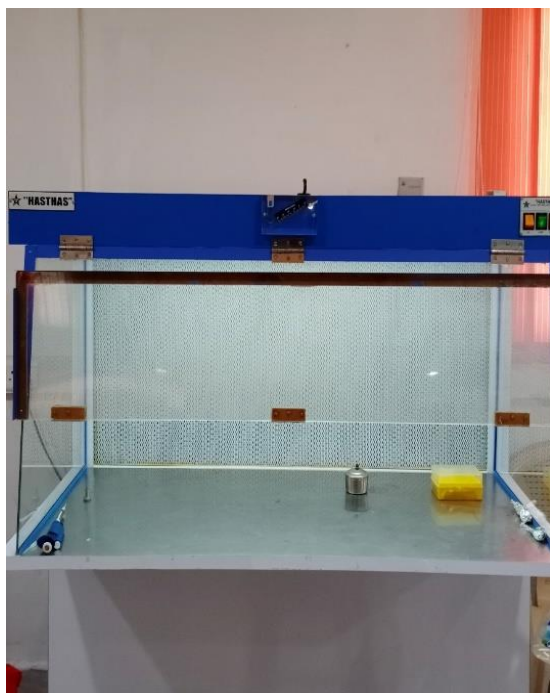


Fig.4.7.a Laminar Air Flow Chamber Fig.4.7.b Inoculating bacteria to media

After inoculation of bacteria, loosely cover the culture with sterile aluminium foil or a cap that is not air tight. Incubate bacterial culture at 37°C for 12-18 hr in a shake incubator.



Fig.4.8. Incubator

After incubation, check for growth, which is characterized by a cloudy appearance in the media.

4.3.5.a. *Pseudomonas*

Pseudomonas is a common genus of bacteria, which can create infections in the body under certain circumstances. There are many different types of *Pseudomonas* bacteria. Only a few types can cause an infection. *Pseudomonas* bacteria tend to live and breed in water, soil, and damp areas. *Pseudomonas* is a genus of Gram negative, Gamma proteo bacteria, belonging to the family *Pseudomonadaceae* and containing 191 validly described species

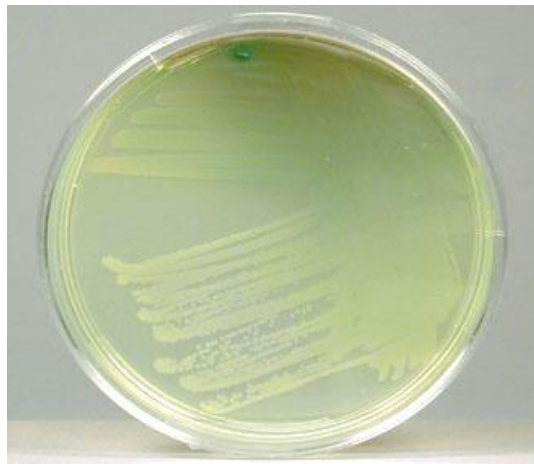


Fig.4.9. Cultured *Pseudomonas*

Functions of *pseudomonas* like other members of the *Pseudomonas* genus, it is known for its metabolic versatility and its ability to colonize a wide range of ecological niche such as rhizosphere, water environments and animal hosts, including humans where it can cause severe infections.[8]

4.3.5.b. *Klebsiella*

Klebsiella is a type of bacteria commonly found in nature. In humans, the bacteria are often present in parts of the digestive tract where they do not generally cause problems. *Klebsiella* is a genus of negative, oxidase negative, rod shaped bacteria with a prominent polysaccharide based capsule. *Klebsiella* species are found everywhere in nature. This is thought to be due to distinct sub lineages developing specific niche adaptations, with associated biochemical adaptations which make them better suited to a particular environment. They can be found in water, soil, plants, insects and other animals including humans.



Fig.4.10. Cultured Klebsiella

Klebsiella organisms can lead to a wide range of disease states, notably pneumonia, urinary tract infections, sepsis, meningitis, diarrhea, peritonitis and soft tissue infections. Klebsiella species have also been implicated in the pathogenesis of ankylosing spondylitis and other spondyloarthropathies.[9]

After the incubation we should check the bacterial growth in the cultured media. The cloudy appearance in the tubes indicating the presence of bacterial growth.

4.4 Collection and Analysis of Water:

One litter of underground water which was used for cultivation of the crops was collected and transported to the laboratory. Water was collected in pre-cleaned polythene bottles as per the standards. Water samples were analysed for fluoride content using Electro chemical probe method. a) Zirconium alizarin method II without distillation. b) Zirconium alizarin method with distillation. and c) Electrochemical probe method

4.5 Collection and Analysis of Grains:

Grains were harvested from the ragi plant from the selected farm itself. Care was taken that no other parts of plant were present. The samples were kept in plastic bags and transported to the laboratory. Samples were washed with distilled water, dried and then crushed into a powder so as to pass through a mesh sieve.

Fluoride analysis of grain done by using the method IS 3025(Part 60) -2013.

CHAPTER 5

RESULTS AND DISCUSSION

5.1 FTIR

The main idea gained from the FTIR analysis is to understand what the meaning of the FTIR spectrum (see example FTIR spectrum in Figure 5.1 & 5.2). The spectrum can result “absorption versus wavenumber” or “transmission versus wavenumber” data. In this paper, we discuss only the “absorption versus wavenumber” curves.

Olefinic (alkene): Vinyl C-H out-of-plane bend 995–985 + 915–890, Vinylidene C-H out-of-plane bend 895–885, trans-C-H out-of-plane bend 970–960, cis-C-H out-of-plane bend 700 (broad).

Aromatic ring (aryl): Aromatic C-H out-of-plane bend 900–670 (several) C-H Monosubstitution (phenyl) 770–730 + 710–690 C-H 1,2-Disubstitution (ortho) 770–735 C-H 1,3-Disubstitution (meta) 810–750 + 900–860 C-H 1,4-Disubstitution (para) 860–800.

Acetylenic(alkyne): Alkyne C-H bend 680–610 Alkyne C-H bend 630 (typical)

Aliphatic organ halogen compound: Aliphatic chloro compounds, C-Cl stretch 800–700 Aliphatic bromo compounds, C-Br stretch 700–600 Aliphatic iodo compounds, C-I stretch 600–500.

Alcohol and hydroxy compound: Primary alcohol, C-O stretch ~1050 Secondary alcohol, C-O stretch ~1100.

Ether and oxy compound: Peroxides, C-O-O- stretch 890–820¹

Simple hetero-oxy compounds,) Phosphorus-oxy compounds:

Aliphatic phosphates (P-O-C stretch) 1050–990, Aromatic phosphates (P-O-C stretch) 1240–1190/995–850.

Silicon-oxy compounds: Organic siloxane or silicone (Si-O-Si) 1095–1075/1055–1020 Organic siloxane or silicone (Si-O-C) 1110–1080.

Thiols and thio-substituted compounds: Thiol or thioether, CH₂-S-(C-S stretch) 710–685 Thioethers, CH₃-S-(C-S stretch) 660–630 Aryl thioethers, ϕ -S-(C-S stretch) 715–670 Disulfides (C-S stretch) 705–570 Disulfides (S-S stretch) 620–600 Aryl disulfides (S-S stretch) 500–430 Polysulfides (S-S stretch) 500–470.

Common inorganic ions: Nitrate ion 1380–1350/840–8155) Phosphate ion 1100–1000 Ammonium ion 3300–3030/1430–13905) Cyanide ion, thiocyanate ion, and related ions 2200–2000 Silicate ion 1100–900.

Note: 1) Normally, it is very weak in the infrared but more characteristic in the Raman spectrum; 2) Lower frequency band because of the conjugated double bond; 3) Higher frequency band characteristic of aldehydes, related with the terminal aldehydic C-H stretch; 4) Asymmetric/symmetric XO₂ stretch (NO₂ and SO₂); 5) Normally, the first absorption is intense and broad, and the second has weak to medium intensity and narrow. The both often exist as multiple band structures, and it may be used to characterize individual compounds.

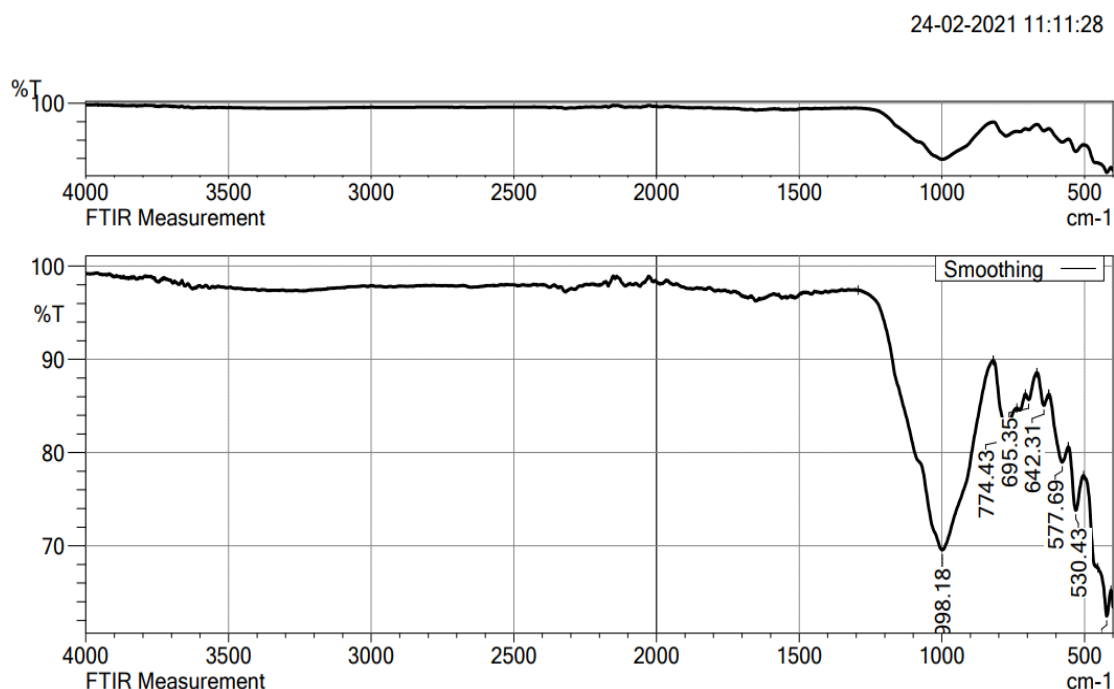


Fig 5.1.a FTIR peak of sample 1

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	422.42	62.47	3.58	453.28	406.99	1611.042	58.005
2	530.43	73.82	5.29	556.47	503.43	1242.964	133.406
3	577.69	78.97	3.39	624.95	556.47	1266.057	131.745
4	642.31	85.06	2.16	666.42	624.95	565.321	43.407
5	695.35	85.70	1.24	706.92	666.42	530.508	21.356
6	774.43	82.27	4.83	819.76	736.82	1259.824	209.203
7	998.18	69.56	23.16	1293.29	819.76	7925.885	4920.708

Table 5.1.b Major peak value of sample 1

2.

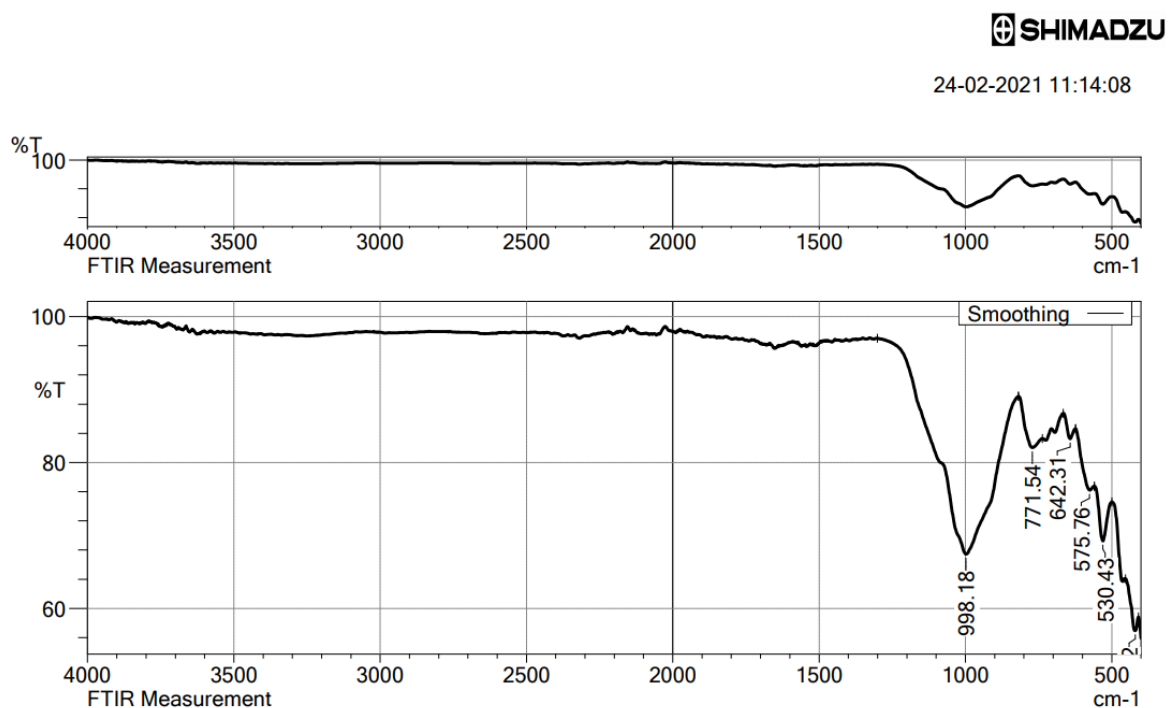


Fig 5.2.a FTIR peak of sample 2

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	419.52	56.95	3.12	453.28	408.92	1773.691	63.323
2	530.43	69.27	6.44	559.37	499.57	1627.065	172.363
3	575.76	76.23	2.52	623.98	559.37	1353.555	106.011
4	642.31	83.26	2.30	665.45	623.98	638.924	45.533
5	771.54	82.06	3.68	818.80	736.82	1304.856	172.046
6	998.18	67.44	24.60	1301.01	818.80	8346.039	4995.766

Table 5.2.b Major peak value of sample 2

5.2 SEM

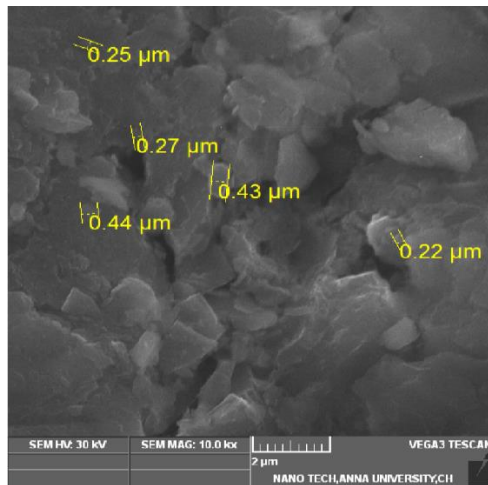


Fig 5.3 Morphology of clay at 2µm

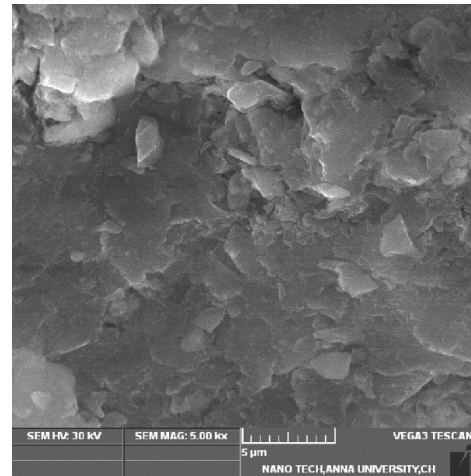


Fig 5.4 SEM image at 5µm

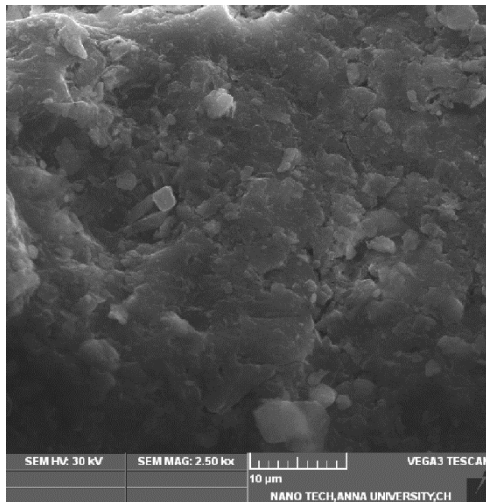


Fig 5.5 SEM image at 10µm

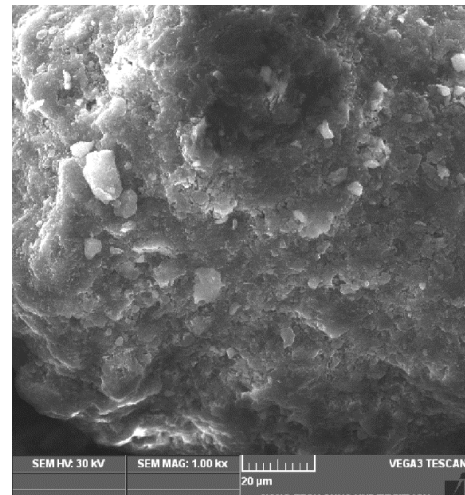


Fig 5.6 SEM image at 20µm

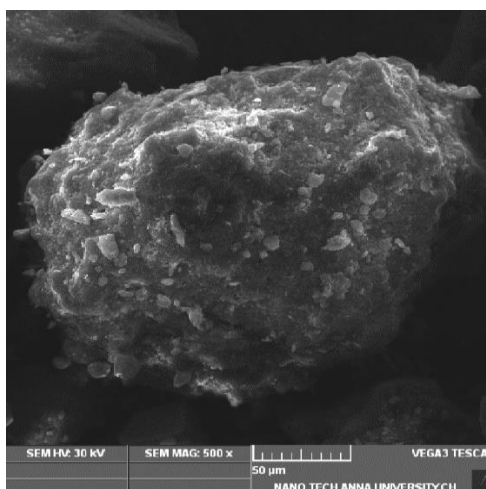


Fig 5.7 SEM image at 50µm

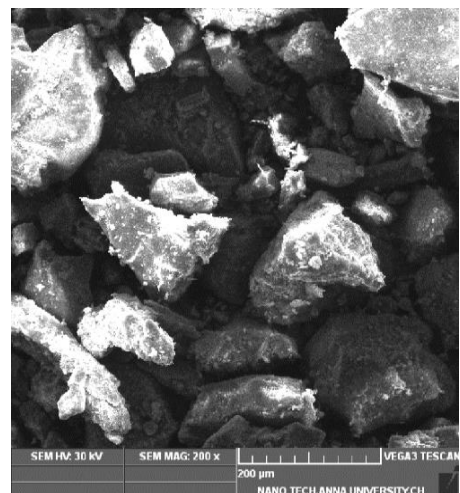


Fig 5.8 SEM image at 200µm

The SEM result reveals that the presence of micro and macro scale materials due to presence of metal or metal oxide in clay it shows brighter SEM images.

5.3 TGA

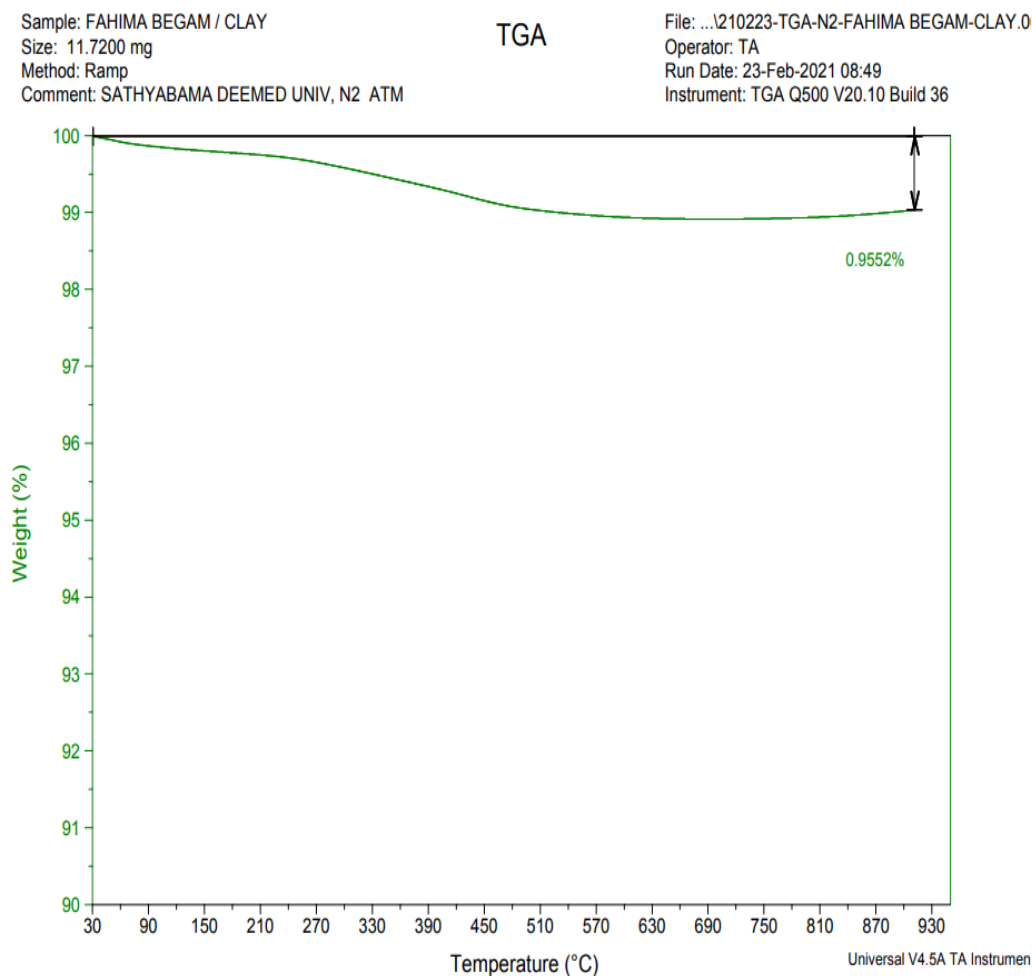


Fig 5.9 TGA of clay sample collected at Kalpakkam

Thermo Gravimetric Analysis of clay sample was done in Nitrogen (N₂) atmosphere. The result was shown in Fig 5.9. It is observed that the clay was thermally highly stable. It reveals that the 90% of the material is stable even above 900 °C.

5.4 SURFACE AREA

Full Report Set

ASAP 2020 V4.04 (V4.04 H)

Unit 1

Serial #: 531

Page 1

Sample: FAHIMA BEGAM / CLAY
Operator: A N
Submitter: SATHYABAMA UNIVERSITY
File: E:\2019\E21-015.SMP

Started: 3/6/2005 5:19:16PM
Completed: 3/7/2005 12:16:55AM
Report Time: 3/7/2005 8:12:12PM
Sample Mass: 0.4569 g
Cold Free Space: 45.6735 cm³
Ambient 25.00 °C
Temperature:
Automatic Degas: Yes

Analysis Adsorptive: N2
Analysis Bath Temp.: -195.608 °C
Thermal Correction: No
Warm Free Space: 15.5330 cm³ Entered
Equilibration Interval: 10 s
Low Pressure Dose: None

Comments: Sample outgassed at 100 C overnight.

Notes:

Summary Report

Surface Area

BET Surface Area: 1.2870 m²/g

Pore Volume

Single point adsorption total pore volume of pores
less than 2013.548 Å width at $p/p^\circ = 0.990307757$: 0.003098 cm³/g

Pore Size

BJH Desorption average pore width (4V/A): 51.604 Å

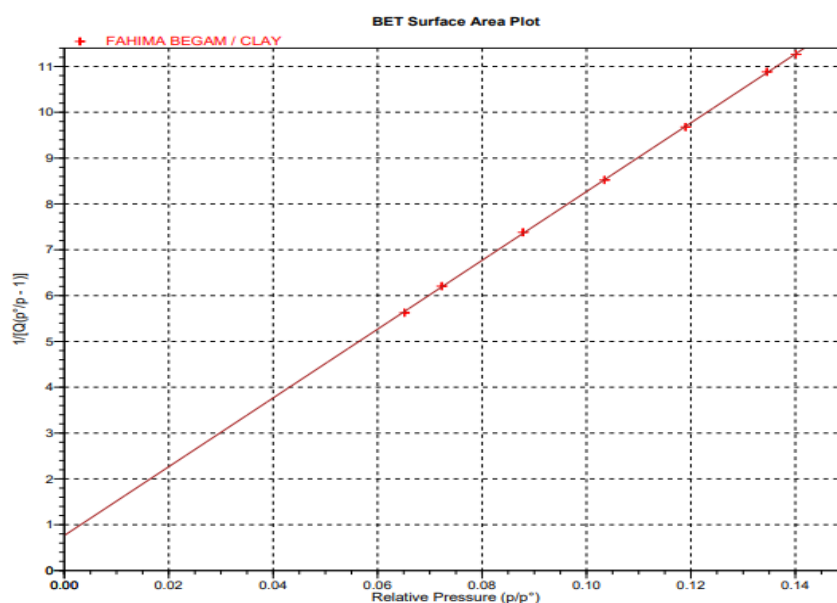


Fig 5.10 BET surface area

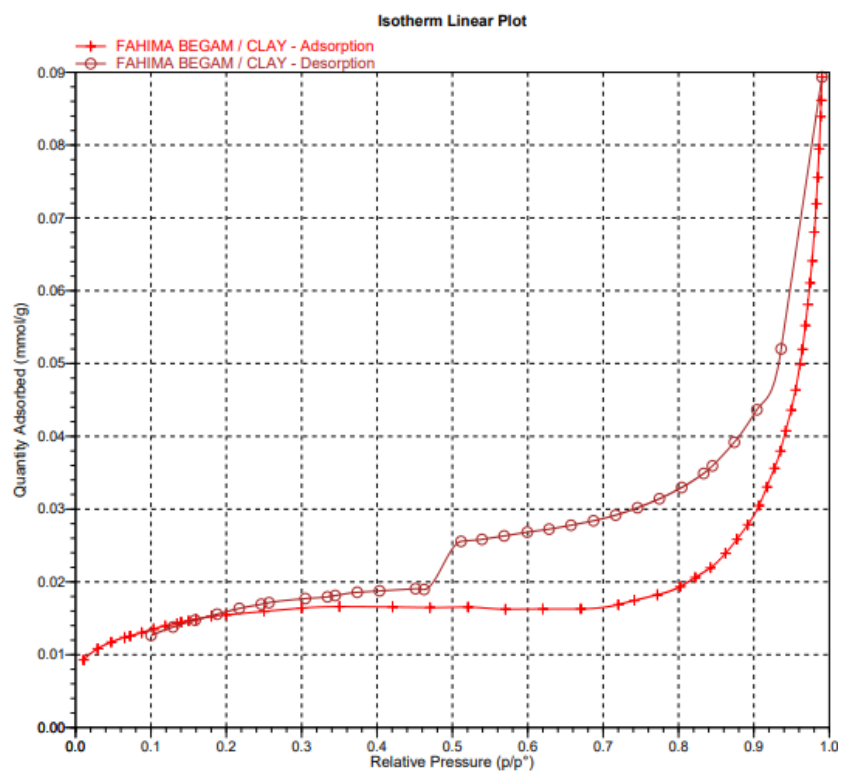


Fig 5.11 Isothermal linear plot

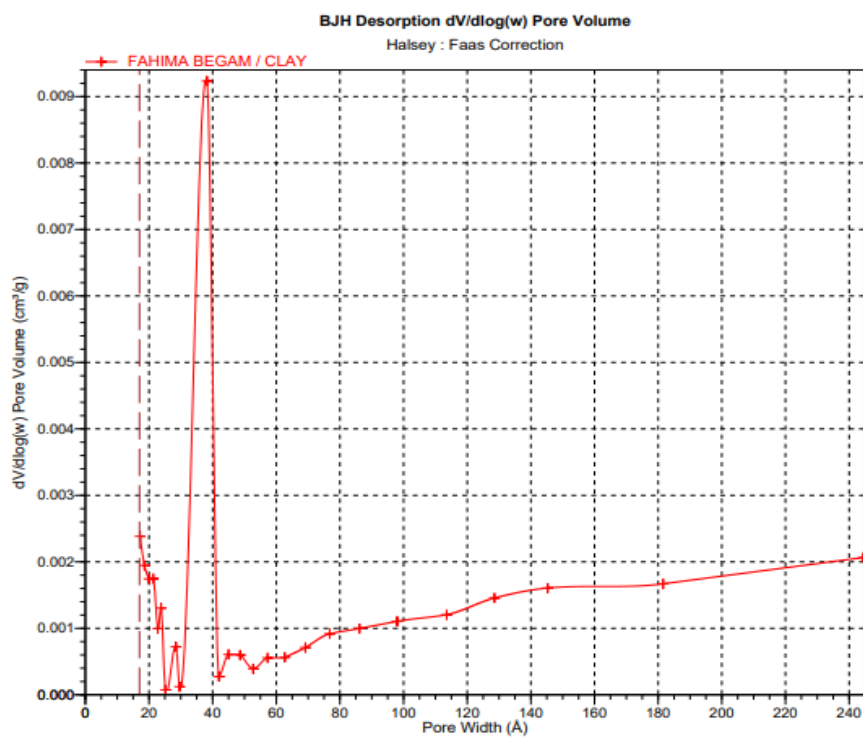


Fig 5.12 Pore volume

The adsorption and desorption isotherm were shown in Fig (5.11 & 5.12). The BET surface area was measured to 1.280 m²/gm though the expected the surface area of clay is less, as particle size decreases, the surface area per unit volume increases.

5.5 XRD

Graphics

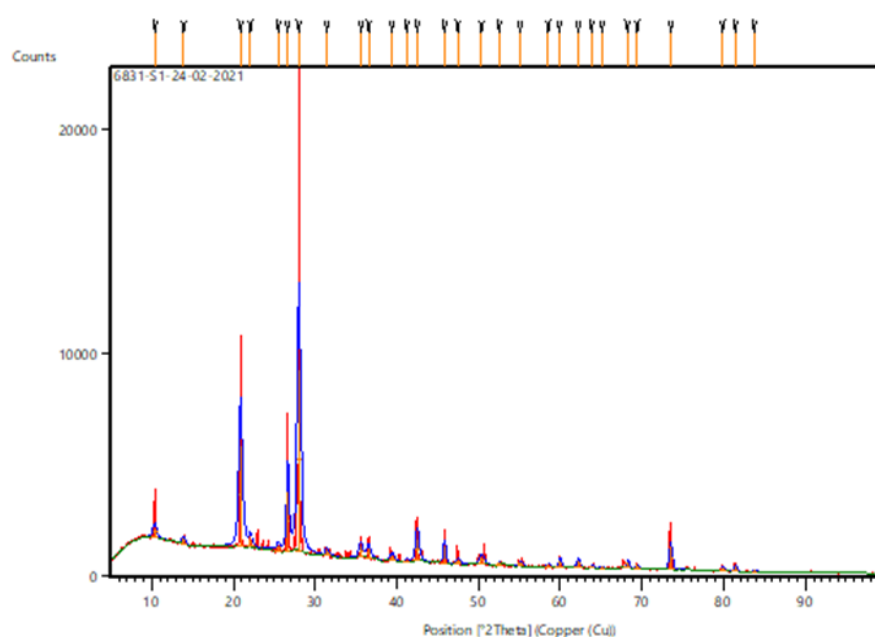


Fig 5.13 XRD value

The presence of above minerals was further tested by XRD studies. XRD is used to determine the mineralogical composition of the raw material components as well as qualitative and quantitative phase analysis of multiphase mixtures. The occurrences of minerals in clay were identified by comparing 'd' values (Selected Powder Diffraction Data for Minerals 1974; Powder Diffraction File Search Manual Minerals 1974). The possible minerals with their 'd' values present in the adsorbents are given in Fig.13. No quantitative estimation phases in these adsorbents have been made but their characterization of XRD patterns indicates the presence of quartz, kaolinite, hematite, illite, and tridymite as the major phases. Further the occurrence of the above minerals in the aforesaid adsorbents was confirmed by FTIR study

5.6 XRF

XRF	
Chemical composition	Weight (%)
SiO ₂	69.90%
Al ₂ O ₃	14.20%
Fe ₂ O ₃	2.39%
MnO	0.05%
MgO	0.61%
CaO	2.77%
Na ₂ O	3.80%
K ₂ O	2.77%
TiO ₂	0.82%
P ₂ O ₅	0.15%
LoI	2.20%
Ba	823ppm
Ce	89ppm
Cr	45ppm
Ga	16ppm
Nb	8ppm
Ni	12ppm
Pb	13ppm
Rb	52ppm
Se	6ppm
Sr	484ppm
Th	9ppm
V	19ppm
Y	25ppm
Zn	29ppm
Zr	148ppm
S	142ppm
Mo	4ppm

Sn	3ppm
W	851ppm
F	2017ppm
Cl	127ppm
CuO ₂	610ppm
Na ₂ CO ₃	99ppm
U	19ppm

Table 5.3 XRF data

It was analysed to know the chemical compositions of the minerals that are present in the clay. The data given in table 5.3 shows that the alumina, calcium oxide and silica oxide are present in major quantities while other minerals are present in trace amounts. This confirms the chemical analysis of clay. Through xrf analysis we find out the amount of fluoride level is extremely high in clay (2017mg/l). similarly, Tungsten Strontium & Copper peroxide is also high.

5.7 OPTICAL DENSITY

SAMPLE	Optical density at 600 nm		ACTUAL GROWTH
	OD ₁	OD ₂	
Blank	0.9024	0.9025	
P cons	1.6219	1.6270	0.7195
p 0.5	1.3938	1.3974	0.4914
p 1 g	1.7142	1.7161	0.8118
K cons	1.7644	1.7715	0.862
k 0.5 g	1.0915	1.0944	0.1891
k 1g	1.2562	1.2621	0.3538

Table 5.4 Optical density value of cultured bacteria

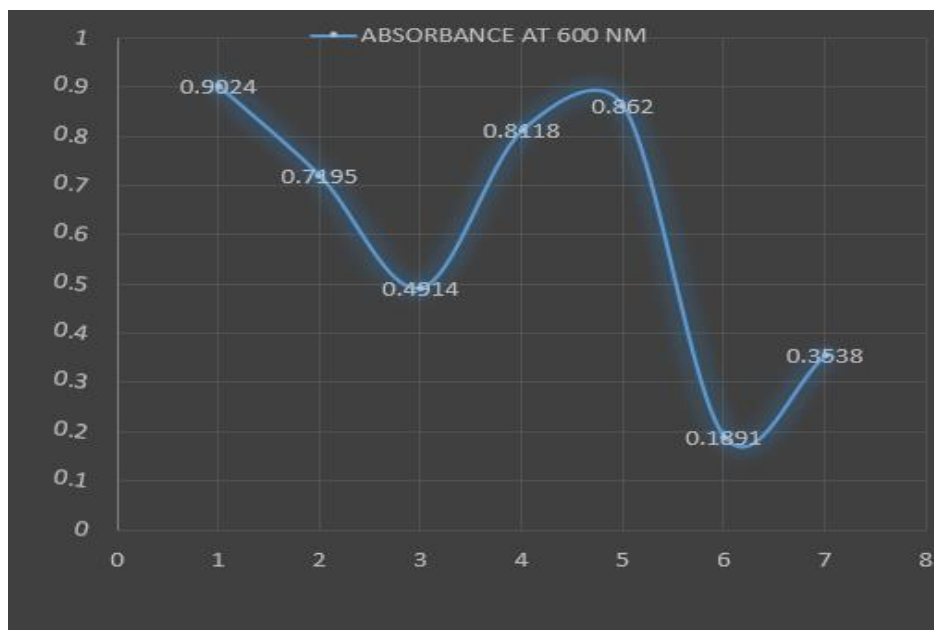


Fig 5.14 Absorbance at 600nm

The above values and graph describe there is a slight growth inhibitor character of soil.

5.8 FLUORIDE DETECTION

S. NO	PARAMETERS	METHOD	UNITS	RESULTS
1	Fluorides	IS 3025(Part 60)-2013 RA:2017	ppm	BQL(LOQ:0.1)

Remarks : BQL: Below quantification Limit ; LOQ : Limit of quantification

Table 5.5 Amount of fluoride in ragi

S. NO	PARAMETERS	METHOD	UNITS	RESULTS
1	Fluorides	IS 3025(Part 60)-2013 RA:2017	ppm	5

Table 5.6 Amount of fluoride in water

The study revealed that high level of fluoride in water (5mg/l), It provides an understanding into the problem of fluorosis among people in the study area,

There was a huge difference in between mean fluoride levels of soil and water ($p < 0.001$) and in between soil and grain ($p < 0.001$); however, difference in between mean fluoride levels of water and grain was found to be non-significant.

CHAPTER 6

SUMMARY AND CONCLUSION

Fluoride plays an important role in human metabolism within the permissible limit. But it is exceeded by many activities mainly by the human activities. High level of fluoride causes major impact in human health. It can replace calcium ion in bones and teeth, which can lead to fluorosis. The highest areas of fluoride contamination of groundwater were identified.

Fluoride level of soil, grains and water should be adjusted to an optimum level. Soil has positive correlation with respect to uptake of fluoride by ragi grains. So, ragi grains with optimum fluoride content should be made available in the commercial markets so that oral and general health can be benefitted.

Endemic fluorosis has remained a major public health problem throughout the world, especially in India. Absolute treatment is difficult; hence prevention and control of fluorosis is important, thus changing the water source, reducing the fluoride concentration of drinking water and decreasing the consumption of fluoride rich food are the main strategies that can effectively diminish the incidence of fluorosis. It is the total amount of Fluoride absorbed in a human body that needs to be considered i.e., the sum of fluoride intake from water, food, and air. Therefore, determining dietary fluoride intake is also useful and important for estimating the retention of fluoride in man. This provided an impetus with the aim to estimate the absorption of fluoride to edible grains from soil and water.

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GEOTAGGED IMAGE



This Geo tagged image is the location from which my clay sample has been collected.