ADDIS ABEBA UNIVERSITY

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MASTERS THESIS

DEVELOPMENT OF NANO ZEOLITE FROM TEXTILE SLUDGE AND INTEGRATING WITH POULTRY MANUER FOR AGRICULTURAL APPLICATION

MAHIDER JENBERU KENU

A THESIS SUBMITTED TO SCHOOL OF CHEMICAL AND BIO ENGINEERING IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE MASTERS OF SCIENCE IN ENVIRONMENTAL ENGINEERING

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DECLARATION

This is to certify that the thesis prepared by Mahider Jenberu Kenu ‘’Development of nano zeolite from textile sludge and integrating with poultry manure for agricultural application’’which is submitted to school of chemical and Bio engineering, Addis Ababa University in partial fulfillment of the requirements for the Degree of Master of Science in Environmental Engineering, complies with the regulations of the university and meets the accepted standards with respect to originality and quality.

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Abstract

The textile industry generates large volumes of sludge during wastewater treatment, containing useful minerals such as silica and alumina. These components make the sludge a raw material for synthesizing zeolite, a crystalline material widely used in environmental applications. This thesis presents an experimental approach to convert textile sludge into Nano-zeolite via alkaline activation followed by microwave synthesis. The sludge, produced Nano-zeolite and composite was characterized using analytical techniques including XRF for elemental composition, XRD for crystal structure, TGA to measure the weight change of sample as it is heated, FTIR for functional group analysis, BET to measure the specific surface area of the produced Nano- zeolite, and CEC of Nano-zeolite from textile sludge, an industrial waste stream, has been achieved using a statistically designed approach. Under the optimized conditions (10 mL/g NaOH, 120°C, and 90 minutes Irradiation time), as high as 72.67% yield of a high-surface-area material (472 m^2/g) could be obtained with FTIR-confirmed aluminosilicate framework vibrations characteristic of Na-X zeolite, crystalline structure XRD of the synthesized material The diffraction peaks were observed at approximately 10.1°, 15.6°, 22.8°, 26.7°, and 31.3° (2θ). These peaks are similar to the characteristic peaks of Nano zeolite X, and the CEC value of the Nano-zeolite is 115cmol+/kg. The Nano zeolite was blended with composted poultry manure at 5%, 10%, and 15% (w/w); all composites were homogenized. The nutrient analysis results show that phosphorus and potassium increased with zeolite content, reaching 60.3 ppm P and 0.84 ppm K and N is as it is 0.31 in the 10% and 15% blends. This enhancement is attributed to the zeolite's high CEC, which retains and stabilizes cations released from manure.

***Key-words**: Cation exchange capacity, Composite preparation, Microwave synthesis, Poultry manure, Textile sludge*

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ABBREVIATION

Abbreviation	Meaning
AAS	Atomic Absorption spectrophotometer
ANOVA	Analysis of Variance
BBD	Box–Behnken Design
BET	Brunauer–Emmett–Teller
BLIP	Bole Lemi Industrial Park
BLTS	Bole Lemi Textile Sludge
CEC	Cation Exchange Capacity
DE	Design Expert®
DI	Deionized
FTIR	Fourier-Transform Infrared Spectroscopy
NaOH	Sodium Hydroxide
NH_4^+	Ammonium Ion
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
TS	Powdered calcined textile sludge
TSA	Textile Sludge Ash
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

CHAPTER ONE INTRODUCTION

1.1Background of the Study

The global textile industry is a cornerstone of the world economy, employing over 60 million people and contributing approximately 3–4% of global GDP. It encompasses fiber production, fabric processing, dyeing, finishing, and garment manufacturing, with major production centers in China, India, Bangladesh, Pakistan, and Turkey. However, its environmental footprint is substantial. The sector is responsible for high levels of water consumption, energy usage, and chemical discharge. It is also known for generating considerable volumes of both liquid effluents and solid waste. According to global estimates, processes such as dyeing and finishing are among the leading contributors to industrial water pollution, accounting for nearly one-fifth of such discharges worldwide;.-(Development & Agency, 1998).

One particularly pressing environmental concern associated with textile manufacturing is the generation of sludge during the wastewater treatment process. This sludge is a byproduct of physical, chemical, and biological treatment methods used to purify contaminated water before discharge or reuse. The quantity and composition of the sludge vary depending on with production scale and treatment technologies used. Typically, it contains a complex mixture of organic substances, synthetic dyes, heavy metals (such as chromium, cadmium, and lead), and inorganic oxides, such as silica (SiO_2) and alumina (Al_2O_3). (Petrov & Michalev, 2012).

Ethiopia's textile production industry has a 3.34% compound annual growth rate (CAGR) from 2021 to 2025, with a 51% increase over the last 5 to 6 years. A major challenge in the textile industry is the increasing scarcity of resources needed for energy production, infrastructure deficiency and Textile waste. (Development & Agency, 1998)(Gejea, 2024).

If improperly managed, textile sludge presents serious environmental and public health risks. When disposed of in landfills, harmful components can leach into the surrounding soil and groundwater, causing long-term contamination. Open dumping or burning may release toxic compounds into the atmosphere, affecting air quality and contributing to respiratory illnesses.

Moreover, the accumulation of persistent organic pollutants (POPs) and toxic metals may disrupt local ecosystems and enter the food chain, creating additional health hazards for both humans and wildlife. (Lateef et al., 2016).

Nano zeolites, which are crystalline porous aluminosilicates, have attracted widespread attention for their remarkable thermal stability, and high surface area. These materials are extensively used in various applications, including catalysis, adsorption, and water treatment. Both natural and synthetic zeolites have shown strong potential for adsorbing and slowly releasing cations. Their use in agriculture as soil amendments or slow-release fertilizer components gained attention for improving nutrient use efficiency and reducing environmental impacts. Removing contaminants from industrial wastewater, including heavy metals and colored compounds. Importantly, synthetic zeolites offer advantages in terms of structural control and purity, as their properties can be fine-tuned through controlled synthesis. (Kianfar & Mahler, 2020).

1.2 Statement of the Problem

Disposing of textile sludge is a significant environmental challenge, especially in developing countries where proper waste management systems are limited or non-existent. Common practices such as landfilling and open dumping are unsustainable and pose serious risks to human health and the environment. Although chemical fertilizers are widely used to boost crop yield, they have several environmental drawbacks. Continuous and excessive application leads to nutrient leaching. Textile sludge poses environmental dangers; it also contains valuable minerals, particularly silica and alumina, which are essential for making nano-zeolites. Nano zeolites are versatile materials that have many uses in environmental and agricultural applications Thus, textile sludge represents an untapped opportunity for resource recovery by converting it into nano-zeolite and mixing it with poultry manure to improve soil. This method offers two benefits: reducing the negative environmental effects of sludge disposal and producing a valuable material with commercial and environmental benefits.

However, the potential of textile sludge for nano-zeolite production has not been thoroughly investigated. Most current research focuses on other industrial wastes, like coal fly ash or municipal solid waste ash. Studies that specifically examine turning textile sludge into nano-zeolite for agricultural use are limited. In addition, the synthesis process requires careful tuning of factors; such as pH, temperature, reaction time, and alkaline solution concentration. Addressing these gaps is key to promoting sustainable waste management and supporting the circular economy. This study suggests a detailed experimental investigation into converting textile sludge into a nano-zeolite composite integrated with poultry manure to enhance nutrient retention and maintain soil health, by doing this, it aims to offer a scientifically valid and practical solution to the problem of textile sludge disposal while helping to develop eco-friendly materials for sludge treatment

1.3 Objectives of the Study

General Objective

The main objective of this study was to synthesize Nano-zeolite from textile sludge through microwave-assisted methods and integrate-it with Poultry Manure to develop a composite fertilizer.

Specific Objective

The specific objectives of this study were

- to synthesize zeolite from pretreated textile sludge using micro wave-assisted method.
- to characterize the properties of the synthesized Nano zeolite X.
- to Evaluate the CEC (cation exchange capacity) of the synthesized Nano-zeolite X
- to Develop poultry manure - Nano zeolite Composite fertilizer

1.4. Significance of the Study

The significance of this study lies in its potential to address two major environmental challenges at once: managing textile sludge waste and developing cost effective, high-performance materials for environmental cleanup. As the textile industry grows worldwide, the amount of sludge from wastewater treatment processes increases, raising environmental and public health concerns. Traditional disposal methods, such as landfilling and incineration, are unsustainable because they are costly, subject to regulatory restrictions, and can release harmful substances.

This study explores the production of Nano-zeolite X from textile sludge as an innovative and sustainable way to recycle waste. Nano-zeolites are well-known for their ability to adsorb, exchange ions, and act as catalysts. Creating zeolite from textile sludge not only keeps hazardous waste out of landfills but also decreases the need for new raw materials and expensive commercial zeolites.

Additionally, the study supports the broader goals of the circular economy by encouraging resource recovery and the industrial reuse of waste materials. The experimental data gathered in this research will provide valuable insights into the synthesis process, material characterization, and slow-release fertilizer properties of sludge-based zeolites. This will contribute to the growing body of scientific literature in this field.

In the long run, the results of this research may help policymakers, environmental agencies, and industry leaders develop sustainable waste management strategies, particularly in areas where textile production is concentrated.

1.5 Scope of the Study

This study examines the production of Nano-zeolite from textile sludge using a microwave-assisted method. It also explores how to create a composite slow-release fertilizer by combining it with composted poultry manure with it. The research includes characterizing the raw materials and optimizing the synthesis parameters, such as temperature, irradiation time and NaOH concentration, using response surface methodology in Design-Expert 13. Thoroughly characterize the Nano-zeolite through XRD, FTIR, BET, XRF, and cation exchange capacity analysis (CEC). The composite is made with different ratios of Nano-zeolite, ranging from 5%, 10%, and 15% by weight. It does not cover field trials, plant growth assessments, and environmental impact studies, all of which are suggested for future research.

CHAPTER TWO: LITRATURE REVIEW

2.1 Introduction to textile industries

The increasing impact of industrial activities on the environment, especially in developing countries, has made sustainable waste management more urgent. The textile sector is a major contributor, generating large amounts of wastewater and solid waste, particularly sludge from wastewater treatment. This sludge is often considered hazardous because it contains dyes, heavy metals, and organic pollutants; that can harm ecosystems and human health if not managed properly. However, it also contains valuable inorganic compounds such as silica and alumina, which can be reused. One promising way to make use of textile sludge is to synthesize Nano-zeolites. These are crystalline, porous aluminosilicate materials commonly used in environmental cleanup, catalysis, and adsorption. While several studies have looked into converting other industrial byproducts, such as fly ash and slag, into zeolites, there is limited research on using textile sludge for this purpose. Given their similar composition, textile sludge is an underused resource with great potential for developing sustainable materials. (Miah, Haque, Khaleque, & Santos, 2023).

2.2 Environmental Impact of the Textile Industry and Sludge Generation

Textile sludge has both organic and inorganic contaminants. Heavy metals like lead, chromium, and cadmium are often found due to the use of dyes and mordant. Other components include synthetic dyes, surfactants, oily residues, and suspended particles. The presence of toxic compounds makes improper sludge disposal a severe environmental issue. In many situations, sludge is dumped in open land or landfills. This allows harmful substances to seep into the surrounding soil and groundwater. The textile industry is important to industrial development, but it is often recognized for its harmful environmental effects. Processes like dyeing, washing, and finishing use large amounts of water and various chemicals. These steps generate significant amount of wastewater that must be treated before being released or reused. A main byproduct of this treatment is a solid residue called textile sludge. This sludge forms during different phases of wastewater purification, including sedimentation, chemical precipitation, biological degradation, and filtration. As impurities are removed from the water, they settle or gather into a thick, semi-solid mass. The properties of this sludge can vary greatly based on the industrial methods used, the chemicals involved, and the types of textile products being made. (Bacakova, Vandrovcova, Kopova, & Jirka, 2018).

Furthermore, without proper control, sludge may contribute to air and water pollution. Decomposing organic matter can emit harmful gases, while persistent chemicals can affect local ecosystems and human health. Because traditional disposal methods are neither sustainable nor cost-effective, research has increasingly turned toward methods that enable the recovery and reuse of useful components from the sludge. One such method is to convert this waste into beneficial materials such as zeolites. This not only helps reduce hazardous waste volume but also offers an opportunity to produce valuable materials for environmental cleanup and industrial processes. (Yoldi, Fuentes-Ordoñez, Korili, & Gil, 2019).

2.3 Chemical Composition and Environmental Concerns of Textile Sludge

Textile sludge is a highly variable and complex material that contains a broad range of chemical constituents, many of which pose serious environmental risks. Its composition depends on several factors, including the types of fibers processed, the chemicals used during dyeing and finishing, and the specific wastewater treatment methods applied. Despite this variability, textile sludge generally contains significant quantities of both organic and inorganic matter, making it both hazardous and potentially valuable .(Petrov & Michalev, 2012).

The inorganic fraction of textile sludge is often composed of various metal oxides, with silicon, aluminum, calcium, iron, and magnesium being among the most commonly detected elements. Among these, silicon dioxide and aluminum oxide are especially relevant due to their role in forming the fundamental structure of zeolite. These compounds contribute to the creation of the three-dimensional, porous network that gives zeolite its unique chemical and physical properties. Other trace elements such as potassium, sodium, and iron may also be present and can influence the characteristics of the synthesized product.(Kianfar & Mahler, 2020)

Organic contaminants are also prevalent in textile sludge. These include residual dyes, surfactants, lubricants, and solvents used during textile processing. In many cases, these compounds are resistant to biological degradation and may interfere with the synthesis of new materials or introduce impurities that limit performance. The sludge may also contain heavy metals such as chromium, lead, cadmium, and copper, introduced through dyeing agents and chemical additives. These metals are particularly concerning because of their toxicity, persistence, and bioaccumulation in living organisms. (Innovations in Textile and Apparel Dyeing, 2024).

From an environmental standpoint, improper disposal of textile sludge can lead to long-term contamination. Heavy metals and synthetic chemicals can leach into surrounding soil and water bodies, disrupting ecosystems and posing health hazards to humans and wildlife. The buildup of organic pollutants in water systems can also lead to eutrophication, oxygen depletion, and the collapse of aquatic life. Furthermore, incinerating or open dumping untreated sludge can release hazardous air pollutants, worsening air quality and contributing to climate change. (Miah et al., 2023).

Due to these risks, textile sludge is commonly classified as industrial hazardous waste. Safe disposal methods are limited, often expensive, and not always effective, particularly in developing regions. As a result, researchers have focused on alternative strategies that not only mitigate environmental harm but also recover useful resources. One promising approach is convert textile sludge into Nano-zeolite, a material valued for its ability to capture and remove contaminants from water.(Pradesh, Pande, & Pradesh, 2017; Pruksawan et al., n.d.).

2.4 Waste Value and Circular Economy Approaches

The growing volume of industrial waste has driven the need for more sustainable waste management strategies, especially in resource-intensive sectors such as textiles. Traditional disposal methods such as landfilling, open dumping, and incineration are no longer considered viable long-term solutions due to their environmental, economic, and health-related drawbacks. As a response to these concerns, the concept of waste valorization has emerged as a key strategy in environmental science and sustainable development. (Liu, 2022).

Waste valorization involves converting discarded materials into usable or economically valuable products by applying scientific or industrial processing techniques. The main objective is not only to reduce the volume of waste that requires disposal but also to extract useful compounds that can be reintegrated into production systems. In the context of textile sludge, this method aims to use the waste as a raw material to generate new, functional products, such as Nano-zeolites. (Murayama, Yamamoto, & Shibata, 2002).

This idea strongly aligns with the circular economy frame work. Unlike the traditional linear model of consumption and disposal, the circular economy encourages the reuse, recycling, and regeneration of materials to keep them in circulation for as long as possible. Integrating waste valorization into industrial operations supports this vision by minimizing environmental impact and reducing reliance on virgin resources. Previous research has shown success in recovering materials from industrial byproducts such as fly ash, furnace slag, and agricultural residues. These materials have been used to produce synthetic zeolites, which have applications in pollution control, catalysis, and water purification. Their utility stems largely from their high silica and alumina content, which are also present in textile sludge. (Al-ghouti, Khan, & Nasser, 2021).

Although textile sludge has received less attention than other waste streams, it holds considerable potential for valorization due to its chemical composition. The successful conversion of sludge into zeolite not only mitigates waste disposal problems but also creates value by producing materials useful for environmental remediation. This dual benefit highlights the importance of exploring sludge-based valorization pathways as part of broader sustainability efforts. By advancing such practices, industries can reduce their ecological footprint and contribute to achieving the Sustainable Development Goals. Furthermore, integrating circular economy principles into waste management systems fosters innovation, resource efficiency, and long-term environmental stewardship .(Nazemi, Shafiei, & Karimi, 2020).

2.5 Nanozeolite–Structure, Properties, and Environmental Applications

The development of Nano-zeolite has further expanded the material potential in agricultural and environmental applications. The particle size of the Nano-zeolite is below 200 nm, which possesses a higher surface area, more active sites, and a faster ion diffusion rate than conventional zeolite. Zeolites are solid, porous materials made from aluminosilicate minerals, where atoms of silicon and aluminum are connected through oxygen bridges, forming a regular and stable crystal lattice. This structure forms interconnected cavities and channels on the molecular scale, allowing zeolites to selectively trap or exchange ions and molecules based on size, shape, and charge. (Miah et al., 2023).the smallest structural unit of zeolite generally described by the formula of

$$M^{X}/_{n} * [(Al2O3)x(SiO2)y] * wH2O \quad \text{---------------} \ (1)$$

Where M represents a hydrogen ion or a metal cation that balances the negative charge of the zeolite framework, x and y are the numbers of $[AlO_4$and $[SiO_4]^{4-}$, n is the valence of the cation, and w is the amount of water present in the zeolite cavities. Moreover, the number of metal cations (e.g., Na^{+}) must match the total negative charge created by the silicon and aluminum ions, which carry charges of 4^{+} and 3^{+}, respectively

Each tetrahedral TO_4 molecules (T represents either Al or Si atoms) are called primary building units (PBUs). These are then combined to form secondary building units (SBUs) that create basic structural polyhedral shapes such as cubes or truncated octahedrons (14-sided)

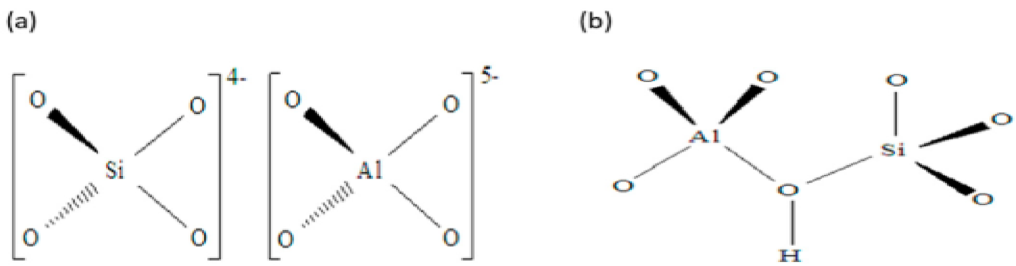


Figure 1Tetrahedral configuration of SiO4 and AlO4 molecules (a) and the corresponding Si-O and Al-O bonds (b) that form unit blocks of a zeolite. (Alsaban et al., 2025)

The framework of zeolite materials carries a net negative charge, which results when aluminum atoms, having a lower valence than silicon, are incorporated into the crystal (Demeke, 2025) structure in place of silicon atoms. To maintain electrical neutrality, positively charged ions such as sodium, potassium, or calcium are located within the pores of the framework. These cations are not fixed and can be replaced with other ions, giving zeolites their high ion-exchange capacity. Additionally, their large surface area, stability at high-temperature stability, and chemical durability make them versatile materials for environmental and industrial technologies. (Eren, Türk, & Arslanoğlu, 2024).

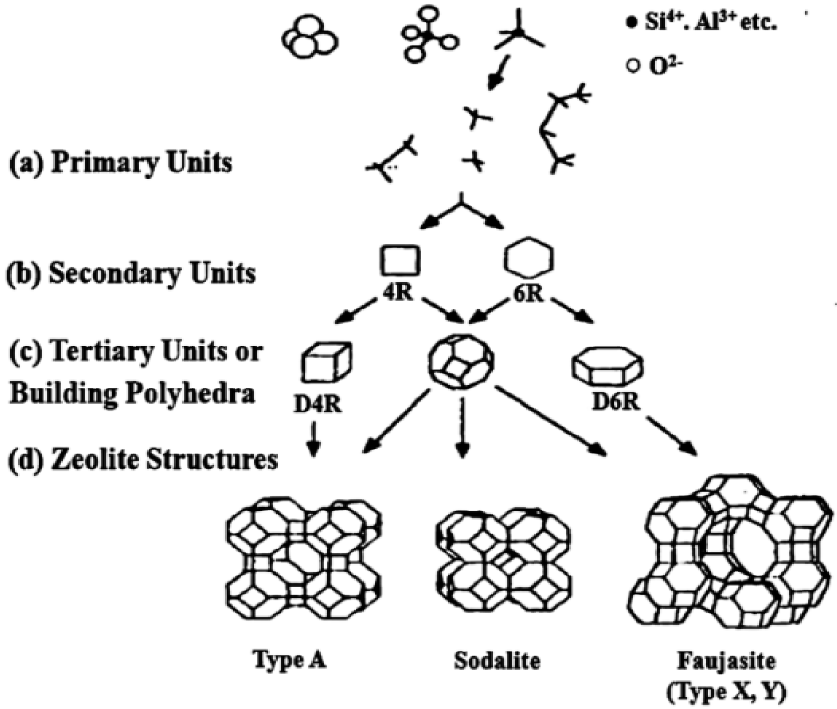


Figure 2 Zeolites occur both in nature and through synthetic processes. (Alsaban et al., 2025)

Natural zeolites form over long periods through the reaction of volcanic ash with alkaline groundwater. These include minerals such as clinoptilolite, mordenite, and chabazite. Each has distinct structural features and environmental applications. Clinoptilolite is widely used for its high ion exchange capacity, including ammonium and heavy metals, making it effective in wastewater treatment and agriculture. Mordenite is valued for its thermal and acid resistance, and is often used in gas separation and catalytic reactions. Chabazite, on the other hand, has a unique crystal geometry that enables it to selectively adsorb gases such as carbon dioxide, making it useful in air purification and carbon capture. Although abundant in nature, these zeolites may require purification or modification to achieve uniformity for industrial use .(Saleh & Al Bahrani, 2023). However, they often require treatment to enhance purity and consistency. Synthetic zeolites, on the other hand, are produced under controlled conditions using either pure chemicals or waste-derived materials like fly ash, slag, or textile sludge. Because their synthesis can be precisely managed, synthetic zeolites often outperform natural ones in terms of uniformity and functional performance. Types such as Zeolite A, X, and Y are widely used in chemical processing, water purification, gas separation, and slow-fertilizer. (Kithome, 1998).

The environmental utility of zeolites stems from their high ability to exchange cations, adsorb organic and inorganic pollutants, and catalyze reactions. They are especially effective at removing heavy metals such as lead, chromium, and cadmium from water systems, making them highly valuable in industrial and municipal wastewater treatment. Additionally, their structure allows them to capture dyes and other colorants from textile effluents, reducing the environmental load of chemical discharges.(Petrov & Michalev, 2012)(Rozhkovskaya, Rajapakse, & Millar, 2021).

The behavior of Nano-zeolites applications is influenced by the chemical composition and physical properties, including pore size, Si/Al ratio, and the type of balancing cations present. For instance, Nano-zeolites with high aluminum content tend to have stronger ion-exchange capabilities but lower thermal stability.(Kianfar & Mahler, 2020).

2.6 Industrial Wastes (Textile) As a Source for Zeolite Production

Industrial wastes are rich in silica and alumina, such as blast furnace slag and rice husk ash, and have also shown potential in zeolite production. Blast furnace slag is a byproduct of the iron and steel industry and contains both alumina and calcium, which can affect the structure and stability of the resulting zeolite. Rice husk ash, produced from the combustion of agricultural waste, is primarily composed of amorphous silica and has been used to produce siliceous zeolites with good adsorption performance. Textile sludge is a byproduct of SiO_2, Al_2O_3, and Fe_2O_3.and organic residue. Waste water treatment contains, in each case, the chemical composition and physical structure of the raw waste material, which influence the type, purity, and surface properties of the resulting zeolite .(Demeke, 2025).

The key to successful zeolite synthesis from industrial waste lies in achieving the correct silica-to-alumina ratio and promoting crystallization under alkaline conditions. This usually involves two main steps: alkaline activation and treatment. During alkaline activation, the raw material is mixed with a strong base, such as NaOH, to dissolve reactive oxides and form a gel-like precursor. The mixture is then aged and heated in a sealed reactor to form crystalline zeolite structures. Additives such as sodium silicate or alumina may be used to adjust the composition, and in some cases, templating agents are added to control pore size and morphology .(Rozhkovskaya et al., 2021).

The most extensively studied precursor for Nano-zeolite synthesis. Fly ash is produced during coal combustion and contains a high proportion of reactive silica and alumina, making it ideal for zeolite formation. Researchers demonstrated that by treating fly ash with sodium hydroxide (NaOH) under hydrothermal conditions, typically involving heating at temperatures between 80 and 200 degrees Celsius for several hours, various types of Nano-zeolite. Such as Zeolite A, X, and P can be synthesized. The efficiency of the process depends on several parameters, including the alkaline solution concentration, reaction temperature, solid-to-liquid ratio, and aging time .(Ojha, Pradhan, & Samanta, 2004).

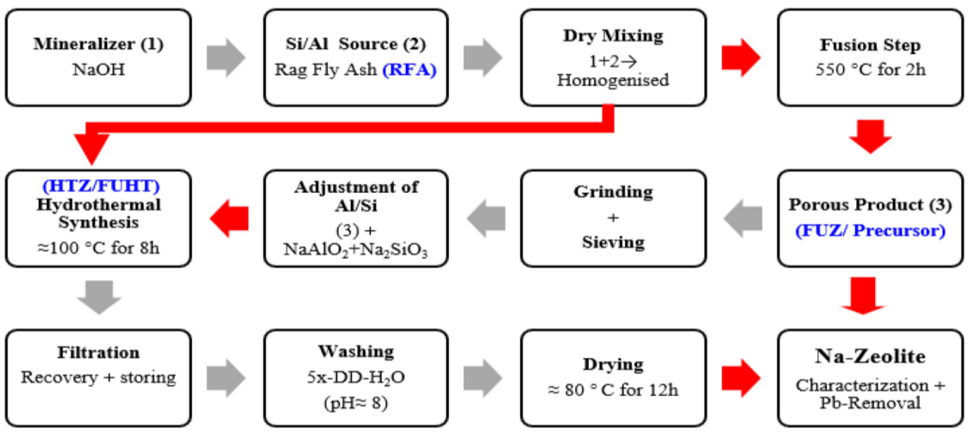


Figure 3 Flow chart process of synthesis by alkali fusion method (Hussain et al., 2021)

2.7 Micro wave assisted Synthesis of Nano zeolite

The synthesis of Nano zeolite from industrial waste has emerged as a sustainable approach to address both environmental pollution and material scarcity. Many industrial byproducts, including fly ash, blast furnace slag, rice husk ash, and red mud, are rich in silica and alumina, key elements required to form the zeolite framework. Rather than disposing of these residues in landfills, they can be chemically transformed into high-value materials through controlled synthesis processes. (Sathupunya, Gulari, & Wongkasemjit, 2003).

Conventional hydrothermal synthesis of zeolite is time-consuming and energy-intensive; it sometimes requires a long crystallization period at high temperatures. Microwave-assisted synthesis method provides rapid uniform heating, resulting in shorter crystallization times, smaller particle size, and higher crystallinity .(Deng, Xu, & Wu, 2016).

Recent developments in zeolite synthesis have focused on optimizing process efficiency and reducing energy consumption. Microwave-assisted synthesis, fusion-assisted techniques, and low-temperature hydrothermal routes are being explored to reduce reaction time and enhance crystal formation. Additionally, the use of waste-derived zeolites is being evaluated for various applications, including heavy metal removal, ammonia adsorption, and catalytic degradation of organic pollutants .(Eren et al., 2024).

2.8 Nano zeolite and poultry manure in agriculture

Natural and synthetic zeolites are widely used in agriculture for their ability to retain ammonium ions and slowly release them to plants. Clinoptilolite and synthetic Na-A zeolite are particularly effective due to their high CEC (150-300MEQ/100g) and surface area (>300 m^2/g) (Mumpton, 1999). Zeolite-amended soils show improved water retention, nutrients availability, and reduced fertilizer requirements. (Lateef et al., 2016)

Poultry manure contains 2-4%N, 1.5-3% P_2O_5, and 1-2% K_2O (on dry weight basis), making it a potent organic fertilizer. However, up to 50% of nitrogen is lost within days of application due to NH_3 volatilization and NO_3^- leaching, especially under alkaline or sandy soil conditions These losses reduce crop yields and contribute to eutrophication and greenhouse gas emissions .(Ruktanonchai, Soottitantawat, Lee, & Tang, 2021; Saleh & Al Bahrani, 2023; Using & Hydrogels, 2024).

2.9 Experimental Approaches for Converting Textile Sludge into Nano Zeolite

Experimental conversion of textile sludge into zeolite typically involves two main steps: alkaline activation and hydrothermal treatment. In the first stage, the sludge is treated with a concentrated solution of a strong base, such as sodium hydroxide (NaOH). This step dissolves the silicate and aluminate components into a reactive gel-like phase that serves as a precursor to zeolite formation. The activation process can be influenced by several factors, including the base concentration, the solid-to-liquid, and the presence of interfering substances such as organic matter or heavy metals.(Belviso, 2020).

Following alkaline activation, the mixture undergoes hydrothermal treatment. This involves heating the reactive gel under controlled temperature and pressure conditions, usually within a sealed autoclave or reactor. Typical synthesis temperatures range from 80°C to 200°C, with reaction times varying from a few hours to several days depending on the targeted zeolite phase. Crystallization occurs during this stage, transforming the amorphous precursor into a well-defined zeolite structure. The parameters of this step, such as temperature, duration, and aging time, must be precisely controlled to ensure high crystallinity and purity of the final product.(Yoldi et al., 2019)(Ramesh & Reddy, 2011).

In some studies, researchers have applied additional pre-treatment steps to improve the quality of the raw textile sludge. These may include drying, calcination, or acid washing. Drying removes excess moisture that could interfere with reaction control. Calcination usually performed at temperatures above 500°C, burns off organic matter and enhances the reactivity of inorganic oxides. Acid washing can reduce the concentration of interfering metal ions or neutralize residual alkaline substances. These steps can improve the overall reactivity of the sludge and increase the likelihood of successful zeolite formation.(Demeke, 2025)

One key challenge in working with textile sludge is the interference from organic dyes and heavy metals. These contaminants can inhibit crystallization or lead to impure products. Therefore, understanding the specific chemical composition of the sludge through preliminary characterization is crucial. Experimental studies often emphasize the need for optimization, testing multiple synthesis conditions to find the most effective parameters for high-quality zeolite production.(Musyoka, Petrik, & Hums, 2012)

2.10 Analytical Techniques for Characterizing Zeolite Materials

The resulting zeolite is usually characterized using a combination of analytical techniques. X-ray diffraction (XRD) is used to confirm the formation of crystalline zeolite phases by comparing the sample's diffraction pattern to known reference structures. X-ray fluorescence (XRF) helps determine the elemental composition, especially the Si/Al ratio, which influences many of the zeolite's functional properties. Fourier-transform infrared spectroscopy (FTIR) provides information about the chemical bonds and functional groups present, while scanning electron microscopy (SEM) reveals the morphology and surface structure of the synthesized material. In some cases, BET surface area analysis is also used to assess the material's porosity and adsorption potential.(Bacakova et al., 2018)

Detailed knowledge of the structural, chemical, and surface characteristics of zeolite is essential when assessing its effectiveness for environmental use, particularly in slow releasing of fertilizer. A range of analytical techniques is used to investigate properties such as crystallinity, elemental composition, porosity, and morphology. These tools are critical for verifying successful synthesis and ensuring that the material meets performance standards for environmental applications.

2.10.1 X-ray Diffraction (XRD)

XRD is one of the most widely used techniques for identifying the crystalline phases present in synthesized zeolites. When a sample is exposed to X-rays, the resulting diffraction pattern reflects its internal structure. By comparing these patterns with reference data (e.g., from JCPDS files), researchers can confirm whether the desired zeolite phase such as zeolite A, X, or Phase formed. The clarity and height of the diffraction peaks help determine how well-ordered the crystal structure is, which reflects the effectiveness of the synthesis process. Highly crystalline structures are usually more effective in applications like ion exchange or adsorption.(Rashid et al., 2020).

2.10.2 X-ray Fluorescence (XRF)

XRF is used to measure the elemental composition of the material, particularly the silicon-to-aluminum. This Si/Al ratio plays a critical role in defining the zeolite’s characteristics. For example, lower ratios indicate greater cation-exchange capacity but lower resistance to acids, while higher ratios suggest better thermal and chemical stability. For sludge-based zeolites, confirming adequate silica and alumina content using XRF is essential before further use .(Bahgaat, Hassan, Melegy, Abd-El kareem, & Mohamed, 2020).

2.10.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is useful for identifying the types of bonds and functional groups present in the zeolite framework. The spectrum often shows characteristic bands for Si–O and Al–O bonds, indicating successful framework formation. It can also detect any residual organic compounds or structural changes resulting from the synthesis process or post-treatment steps. This information is especially valuable when assessing the purity and completeness of the sludge-to-zeolite conversion .(Hussain et al., 2021).

2.10.4 Scanning Electron Microscopy (SEM)

SEM is used to examine the physical appearance of the zeolite crystals. It provides high-resolution images that reveal the size, shape, and surface texture of the particles. Well-defined and uniform crystals usually indicate successful crystallization, while irregular shapes may suggest incomplete synthesis or contamination. SEM is particularly useful for spotting non-zeolitic materials or morphological defects .(Mohamed, Mkhalid, & Barakat, 2015).

2.10.5 Brunauer–Emmett–Teller (BET) Surface Area Analysis

BET analysis evaluates the specific surface area and porosity of a sample by measuring how much nitrogen gas the material can adsorb. These characteristics are essential when the zeolite is intended for adsorption applications. Materials with larger surface areas typically offer improved ability to capture and retain pollutants due to increased active sites. Although BET is not always required for basic characterization, it becomes highly relevant when testing performance in dye or metal ion adsorption .(Eren et al., 2024).

2.10.6 Thermo gravimetric Analysis (TGA)

TGA can be used to evaluate thermal resilience and detect weight loss due to water or volatile substances within the zeolite structure. This technique tracks how the sample's weight changes with increasing temperature, helping identify moisture content, organic residues, and decomposition points. TGA is especially beneficial in assessing the success of pre-treatment and predicting material performance under heat-intensive applications .(Lateef et al., 2016).

Together, these analytical techniques provide a comprehensive understanding of the zeolite's structure and function. For zeolites synthesized from textile sludge, they ensure that the product is suitable for its intended environmental applications. Accurate and consistent characterization not only validates the synthesis process but also builds confidence in the material's reliability for industrial-scale use in adsorption, catalysis, ion exchange and environmental uses.

2.11Cation exchange capacity and its importance

CEC is a key parameter to measure the ability of a material to retain and exchange positively charged ion s between its surface and the soil solution, Material with a high CEC can absorb NH_4^+, K^+, CA^{2+} and Mg $^{2+.}$ In environmental Application especially in fertilizer a material with high CEC means having better nutrient holding and slow realizing properties. Zeolite, due to their negatively charged frame works, naturally encountered as high CEC values which further enhanced when synthesized at nano scale. (Bahgaat et al., 2020)(Hussain et al., 2021)(Top & Vapur, 2020)

2.12Chemical fertlizer versus nanofertlizer

Table 1 Key differentiation between chemical fertilizers versus Nano fertilizer (De Smedt, Someus, & Spanoghe, 2015)

Features	**Chemical fertilizer**	**Nano fertilizer**
Particle size	1-4mm	1-100nm
Nutrient use efficiency	Low (30-50%)	High (>80-85%)
Release mode	Rapid (4-10days)	Controlled/slow(40-60days)
Absorption	It's with soil solution	Direct with root pores
Environmental risk	High leaching emission/run off risk	Low leaching emission /run off risk

2.13 Research Gaps Identified from Literature

The issues identified in the current literature included the limited utilization of textile sludge as a raw material, lack of standardized synthesis procedures, insufficient studies on toxicity and leaching behavior, and inadequate information on economic feasibility and scalability.

To address these challenges, this study focuses on the development of Nano zeolite from textile sludge by microwave synthesis which shows advantages such as reduced processing time and improved efficiency. The synthesized Nano-zeolite is then evaluated for its cation exchange capacity (CEC) to determine its effectiveness in nutrient adsorption and exchange.

Furthermore, a composite material combining Nano-zeolite and poultry manure is prepared to enhance nutrient availability and retention. Comprehensive characterization techniques are employed to analyze the structural and chemical properties of the materials.

Over all, this study contributes to addressing the existing research gaps and provides a pathway toward environmentally friendly and economically viable fertilizer development.

CHAPTER THREE :MATERIAL AND METHODS

3.1 MATERIALS AND SAMPLE COLLECTION

3.1.1 Textile Sludge

Samples of textile sludge were collected from the Bole Lemi Industrial Park in January and March 2025. The location of Bole Lemi Industrial park is in the capital city of Ethiopia, Addis Ababa, where Bole International Airport is not far away. Bole Lemi Industrial park is 327 Ha park, focused on clothing, leather goods and textiles. The Bole Lemi Industrial Park showed in Figure 4 (A) includes a fully operational Water Treatment Plant (WTP) with a daily treatment capacity of 1,500 m^3. This WTP is equipped to handle all industrial liquid waste produced by tenant companies at the facility and undergoes physical, chemical, and biological treatment processes. Currently, the WTP processes an average of 300 m^3 of industrial effluent each day from these tenant companies. The quality of the liquid waste is monitored both before and after treatment by the WTP laboratory to ensure compliance with the prescribed standards for treated effluent. The filtration sludge generated from the WTP's operations is securely stored within the facility for future disposal at designated landfill sites in the future. (Project & May, 2018)

A) Bole lemi industrial park B) Textile sludge sample

Figure 4 Collected textile sludge sample generated from bole Lemi industrial park (BLIP)

3.1.2 Poultry Manure

Fresh poultry manure (mixture of excreta and bedding material) was collected from a local commercial poultry farm. It was air-dried, homogenized, and stored in a cool, dry place until composting.

3.1.3 Chemicals

All chemicals used were of analytical grade:

- Sodium hydroxide (NaOH, ≥98%) – Merck:-

Used as an alkaline activating agent to dissolve silica and alumina from textile sludge and help the Nano-zeolite structure form during synthesis.

- Deionized (DI) water (18.2 MΩ·cm resistivity)

Used as a solvent and washing medium to ensure a contamination-free environment, maintain reaction purity, and remove residual impurities after synthesis.

3.1.4 Equipment

The equipment used in this study are selected based on their role in the synthesis and analysis of zeolite derived from textile sludge Hotplate, Beaker, Analytical Balance, Grinder (RRH-200 grinder) Hot plate, oven, muffle furnace, P^H meter, and flask, micro wave digestion system (ETHOS ONE), vibratory mixer, spectro photo meter, shaker, centrifuge, XRD, XRF (Thermoscientific Niton™ XL2), Thermo Fisher, FT-IR (PHY-FTIR-003), BET (Horiba SA-9600) and TGA.

3.2 METHOD

3.2.1 Experimental frame-work

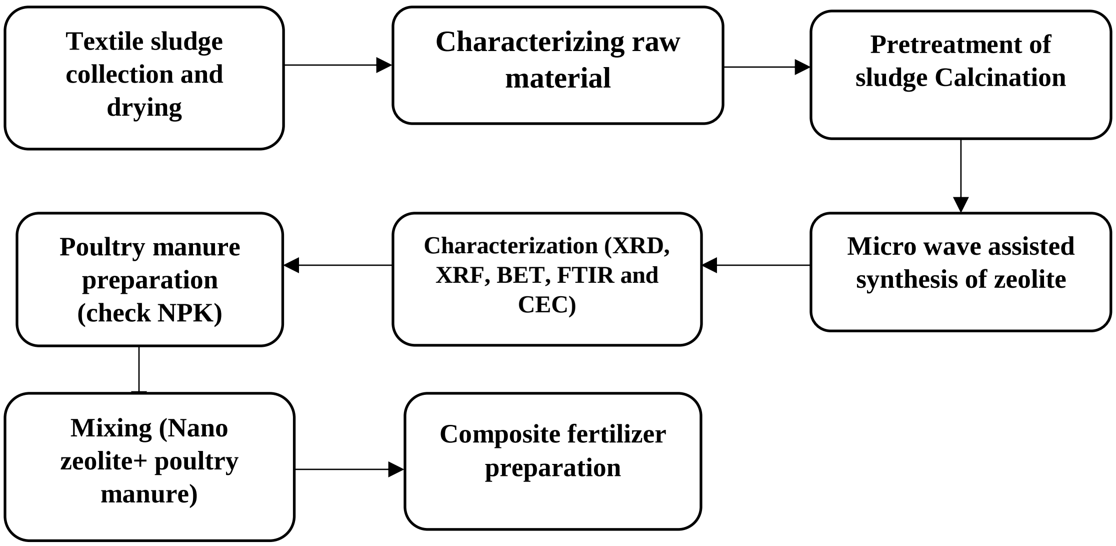


Figure 5 the general schematic diagram of the study presented in flow chart

3.2.2 Feedstock Samples Characterization

3.2.2.1Proximate analysis

Ash Content (AC)

The ash content of the sample was determined according to NREL-LAB. Two grams of oven-dried dried sample were placed in a crucible *(W2*). Then the crucible containing the sample was placed in a muffle furnace (model, manufacturer) and heated at 600^oc for 2hours, or until all the carbon was removed .The crucible was removed the muffle furnace and cooled to room temperature in a desiccator. Finally, the weight of ash (*W1*) was recorded. and the ash content percentage was calculated using the equation .(Wei et al., 2018).

$$Ash\ content = \frac{W2}{W1} * 10 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots Equation(2)$$

Where:

$W1$ =*The weight of ash in grams*

$W2$ = *The weight of moisture free test sample in grams*

3.2.2.2 Elemental Analysis of BLIP Sludge (XRF)

The major oxides' chemical composition of the BLTS sample was analyzed using an X-ray fluorescence spectrometer (Niton™ XL2 XRF, Thermo Fisher) prior to synthesis conducted in the CCIIRDC laboratory. The sun-dried samples were subsequently placed in an oven for further drying. This process took two hours and utilized a DHG-9055 model. They were then ground using an RRH-200 grinder. Following this, the samples were sifted through a 120-mesh sieve. All these steps prepared them for the upcoming analysis.(Yoldi et al., 2019).

Table 2Elemental Analysis of BLIP Sludge (XRF)

No	Parameters	Units	Methods
1.	Silicon (Si)	%	XRF
2.	Aluminum (Al)	%	XRF
3.	Calcium (Ca)	%	XRF
4.	Iron (Fe)	%	XRF
5.	Lead (Pb)	%	XRF

3.2.2.3 XRD Analysis of BLIP Sludge

X-ray diffraction (XRD) is crucial before commencing any experiments, particularly when the goal is to convert textile sludge into Nano-zeolite. This technique reveals the exact crystalline phases present in the material, such as calcite or quartz. Identifying these phases clarifies whether the feedstock is suitable and what type of pre-treatment it may need. Without XRD, one might continue with materials containing problematic phases. For example, calcite releases Ca^{2+} ions that can interfere with zeolite synthesis. Quartz, on the other hand, remains inert and requires activation to transform into useful silica. In either case, it can lead to unsuccessful syntheses or unwanted byproducts. XRD also provides a reliable foundation, allowing for later verification of whether the zeolite was formed properly. It aids in making informed decisions concerning calcination, acid washing, alkali concentrations, and the overall reaction conditions.(Alaya-Ibrahim, Kovo, Abdulkareem, Adeniyi, & Yahya, 2020; Bahgaat et al., 2020; Top & Vapur, 2020)

3.2.2.4 TGA Analysis of BLIP Sludge

Thermogravic analysis measures the material's thermal behavior and compositional breakdown as a function of temperature, exposing important details like moisture content, organic matter (such as fibers, dyes, and polymers), inorganic carbonates (like calcite), and residual ash.(Sadiq et al., 2025)

3.2.3 Pretreatment of Sludge

The raw textile sludge collected from the Bole Lemi Textile Park was subjected to a two-step pretreatment process to enhance its reactivity and remove organic interferes prior to zeolite synthesis

Drying and Size Reduction:

The sludge was initially dried in the open air at room temperature for 72 hours, and then subjected to oven drying at 105°C for 24 hours to reach a stable weight and eliminate remaining moisture. After drying, the material was ground into a fine powder with a mechanical grinder and passed through an 80-µm sieve to achieve a consistent particle size fraction of less than 200 µm shown in Figure 6, ensuring uniformity and enhanced reaction kinetics during synthesis.(Eren et al., 2024)

A) Dried sludge grinded in grinder machine B) meshed textile sludge

Figure 6 Grinded powdered textile sludge ready for calcination

Calcination

Approximately 100 g portions of the dried powdered sludge were placed in ceramic crucibles and calcined in a muffle furnace at 600°C for 2 hours (with a ramp rate of 10°C/min) to combust organic matter (including dyes, surfactants, and pathogens) and concentrate inorganic oxides—particularly SiO_2 and Al_2O_3 which serve as the primary precursors for zeolite formation. The resulting product, referred to as Textile Sludge Ash (TSA), was stored in a desiccator seen in Figure 7 for subsequent use in microwave-assisted synthesis. (Hussain et al., 2021).

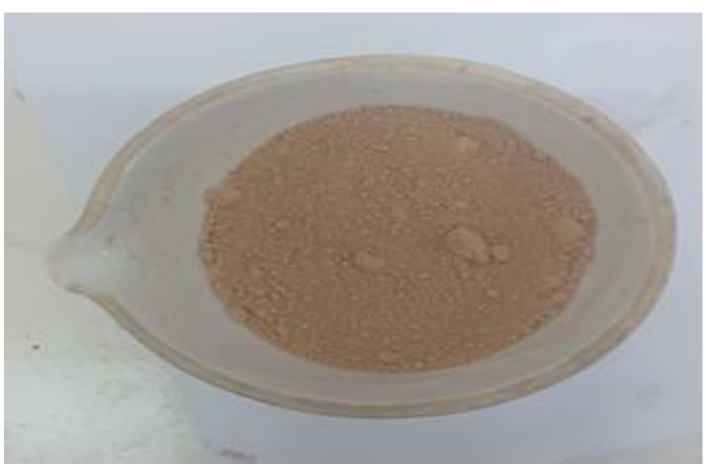


Figure 7 powdered textile sludge after calcination at 600 °C for 2 hours

3.2.4 Experimental Design

The Repose Surface Methodology (RSM) of the Box–Behnken Design (BBD) was applied in the Design of Experiments (DoE) using Design Expert® software V13 (DE). BBD was selected to study the influence of each factor and their interactions. The input factors were NaOH to calcined Bole Lemi textile sludge (TS) ratio (A), Temperature (B), and Irradiation time (C). This resulted in 17 runs, the independent variables were ranged as shown in Table 2. The optimal response of biochar was determined by examining the correlations between the responses (Nano zeolite yield and pH) and the quantitative experimental variables (mixing ratio, irradiation time, and temperature).

Using the DE software, the best model is determined based on F-value, p-value, adjusted and predicted R2 (within 0.2 differences). NaOH to bole Lemi Calcined textile sludge (TS) ratio (A), temperature (B), and irradiation time (C), 2-factor interactions (2FI), and quadratic effects were included in the model if the p-value was < 0.05. To maintain the model hierarchy, factors with p-values > 0.05 were included if their 2FI or quadratic effect was significant (Design-Expert Version 13, 2021). (Saleh & Al Bahrani, 2023)

Table 3Experimental conditions

Variable name	**Unit**	**Factor (X)**	**Level**		
			Minimum	**Middle**	**Maximum**
TS :NaOH	mL/g	A	1:5	1:10	1:15
Temprature	^{0}c	B	100	120	140
Time(Irradiation time)	min	C	60	90	120

3.2.5 NanoZeolite production (Microwave-Assisted Synthesis Procedure)

The synthesis of Nano-zeolite from Calcined Textile Sludge (TS) was carried out using a microwave- method, which enhanced the rate of nucleation and decreased the crystallization time during the synthesis process, offering rapid, uniform heating and reduced reaction time compared to conventional hydrothermal techniques .(Eren et al., 2024).

Slurry Preparation

A mass of 5.0 g of calcined textile sludge was mixed with a predetermined volume of sodium hydroxide (NaOH) solution (3M) The mixture was stirred for 30 minutes at room temperature to ensure homogeneity and initiate alkaline dissolution of silica alumina .(Eren et al., 2024).

Microwave Irradiation

The slurry was transferred into a microwave digestion vessel and subjected to microwave irradiation under designated conditions. TS: NaOH Temperature and Time these parameters were defined by the Box-Behnken Design in Design-Expert 13. Microwave energy promotes rapid nucleation and crystallization of zeolite phases by enabling instantaneous and volumetric heating. (Sadiq et al., 2025)The experiment was held at Addis Ababa University, Addis Ababa, Ethiopia. All runs in this research was conducted in a batch irradiation process.

3.2.6 Nano-zeolite yield calculation and characterization techniques

3.2.6.1 Zeolite Yield

A digital balance was used to weigh the precursors and feedstock both before and after they were charred. The weight was measured in order to calculate the yield of zeolite produced at various temperatures, mixing ratios, and residence Time. The following formula can be used to determine the biochar yield. (Makgabutlane, Nthunya, Nxumalo, Musyoka, & Mhlanga, 2020)

$$Zeolite\ Yield = \frac{Mass\ of\ dried\ nano\ zeolite(gram)}{Mass\ of\ TSA\ used\ (gram)} * 100 \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Zeolite Characterization

The physical and chemical properties of the produced zeolite samples were characterized using standard test methods.

3.2.6.2 Elemental analysis of the samples

The elemental analysis of the zeolite samples at optimum value was performed using Thermo scientific Niton XL2 plus analyzer at the Chemical Construction Industry Research and Development Center (CCIIRDC). The samples were dried in DHG -9055 oven at 105^{o}C for 2 hours to remove moisture, followed by grinding with RRH-200 grinder. The samples were then sieved and homogenized using a 120 Mesh sieve to prepare for elemental analysis.

3.2.6.3 FTIR and Brunauer-Emmett-Teller (BET) Analysis

FTIR spectroscopy was used to characterize the chemical composition and functional groups present in the sample. Approximately 1 mg of the selected zeolite samples was mixed with 100 mg of KBr (potassium bromide) powder and pressed into a pellet using a hydraulic press performed at Addis Ababa Science and Technology University (AASTU).

3.2.6.4 Brunauer-Emmett-Teller (BET) Analysis

The specific surface area of the selected Nano zeolite samples was determined by Brunauer-Emmett-Teller (BET) method using a Horiba SA- 9600 series surface area analyzer at Addis Ababa Science and Technology University. It involves the adsorption of gas molecules (typically nitrogen) on the surface of the sample, and the subsequent analysis of the adsorption-desorption isotherm to calculate the specific surface area .(Oliveira et al., 2023).

3.2.6.5 X-ray diffraction (XRD) Analysis

XRD was used as a rapid, noncontact and nondestructive technique that provides detailed Information about the crystallographic structure, chemical composition, and physical properties of Nano materials. Analyzing the structure of nanomaterials can be done effectively by X-ray diffraction (XRD). The interaction of X-rays with a substance causes its atoms to scatter the radiation. When x-rays are released and interact each other in crystalline material, the atoms' regular organization can be revealed through diffraction pattern that indicates the arrangement of atoms in the material. A powder XRD analysis was performed using a SHIMANDUZ XRD 7000S diffractometer (SHIMANDUZ, Japan). An x-ray tube, sample holder and x-ray detector are the three basic components of an x-ray diffractometer. A cathode ray tube produces x-rays by burning a filament to produce electrons, providing a voltage to accelerate the electrons towards a target and then hitting the target material electrons. Characteristic X-ray spectra are created when electrons possess enough energy to dislodge the target material's inner shell.(Yoldi et al., 2019)

Origin software is then used to turn the data into an XRD graph, and the Scherer equation is used to determine the particle's size.

The Scherrer equation relates the size of crystallites in a material to the broadening of a peak in an X-ray diffraction pattern:

$$D = K\lambda/\beta cos\theta \quad \ldots\ldots \quad \textbf{(4)}$$

Where:

- D is the crystallite size
- K is the shape factor (typically 0.9)
- λ is wavelength of an x-ray
- β is peak width at half maximum intensity
- λ is the X-ray wavelength
- β is the peak width at half maximum intensity
- θ is the Bragg angle by measuring the peak width, the average crystallite size in a nanomaterial can be estimated and smaller crystallites lead to broader peaks.

3.2.6.6 Cation Exchange Capacity (CEC)

The Cation exchange capacity of zeolite was determined using the NaCl Replacement and then distillation method at Oromia Engineering Corporations. (Makgabutlane et al., 2020).

3.2.6.7 pH and EC analysis Test

To determine the pH and EC of the experimental samples, each sample was introduced independently to distilled water at a ratio of 1:20. Five grams of Nano-zeolite and fifty milliliters of distilled water were added to the conical flask. After shaking the mixture for an hour in a mechanical shaker, the pH was measured using a digital pH meter .(Eren et al., 2024).

3.2.7 Post-Synthesis Treatment

Cooling

After irradiation, the product was allowed to cool to room temperature. Filtration was used to recover the solid, and excess alkali and soluble salts were removed by repeatedly washing the filtrate with deionized (DI) water until the pH was appropriate .(Hussain et al., 2021)(Sadiq et al., 2025).

Drying

The washed solid was dried in an oven at 105°C for 12 hours to achieve a consistent weight. The fine powder was then stored in a desiccator for calculating yield, Characterization and composite formation. This technique aligns with green chemistry principles by drastically reducing synthesis time (from up to 48 hours to less than 2 hours), improving crystallinity, and increasing energy efficiency .(Makgabutlane et al., 2020).

3.2.8 Data analysis

Statistical analysis was carried out using Design Expert 13, and Microsoft Excel. A three way analysis of variance (ANOVA) was used to evaluate significant differences among mean values; the F value ($p < 0.05$) was tested. All samples were analyzed in triplicate ($n=3$). The results were presented as mean values plus or minus the corresponding standard deviation. A variety of tools and functions within these software programs were used to perform trend analysis, calculate descriptive statistics, and generate visualizations, including graphs and charts, to support the interpretation of the findings.

3.2.9 Composite Fertilizer Preparation

To develop a slow-release fertilizer formulation, the optimized Nano-zeolite (obtained from Stage synthesized) was physically integrated with poultry manure at three different ratios by weight 5% Nano-zeolite + 95% poultry manure (w/w),10% Nano-zeolite + 90% poultry manure (w/w), and 15% Nano-zeolite + 85% poultry manure (w/w).The poultry manure was first composted aerobically for 21 days to stabilize organic matter, reduce pathogens, and minimize the potential for ammonia volatilization. After composting, it was air-dried, ground, and sieved (<2 mm) to ensure homogeneity. For each blend, the predetermined amounts of Nano-zeolite and composted manure were mixed thoroughly in a mechanical blender for 15 minutes to achieve a uniform composite. The resulting mixtures were stored in sealed polyethylene bags at room temperature until use in nutrient release studies. (Saleh & Al Bahrani, 2023)

This formulation strategy leverages the high cation exchange capacity (CEC) of Nano-zeolite to retain ammonium (NH_4^+), potassium (K^+), and other cations released from poultry manure, thereby reducing nutrient loss and enabling gradual release—key characteristics of composite fertilizer. Addis Ababa University and MIDI, Ethiopia, Addis Ababa. All runs in this research were conducted in a Batch micro wave synthesis process.

CHAPTER FOUR :RESULTS AND DISCUSSIONS

4.1CHARACTERIZING THE RAW SLUDGE

4.1.1 XRF analysis of raw sludge

The spectra studied are predominantly composed of silicon and aluminum with trace levels of other elements shown in Figure 8, including heavy metals (Ag, Cd, and Br).The two overlapping spectra provide a comprehensive view of the elemental makeup across various energy levels. (Ojha et al., 2004).

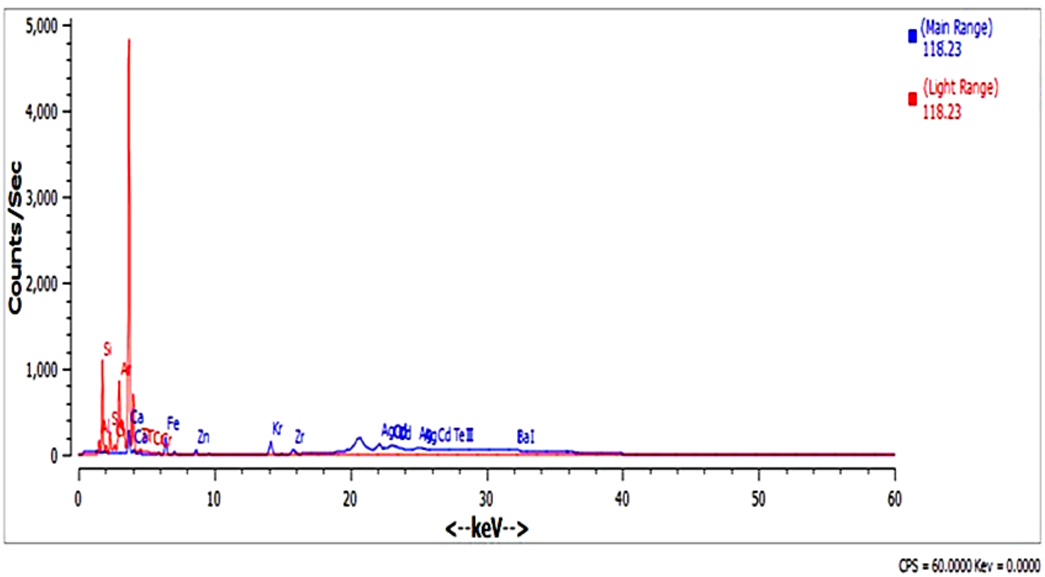


Figure 8 XRF spectrum of raw textile sludge generated from BLIP

4.1.2 XRD analysis of BLIP sludge

The XRD of BLIP indicates that the sample is a combination of two crystalline minerals: Calcite ($CaCO_3$) is the predominant component, accounting for 75.7% of the sample. Quartz (SiO_2) is the minor component, accounting for 24.3% of the sample shown in Figure 9. The excellent Rwp value (11.1%) and near-zero difference plot show that the model accurately describes the sample composition. This type of result is prevalent in geological materials, such as limestone containing calcite and silica.(Sathupunya et al., 2003)

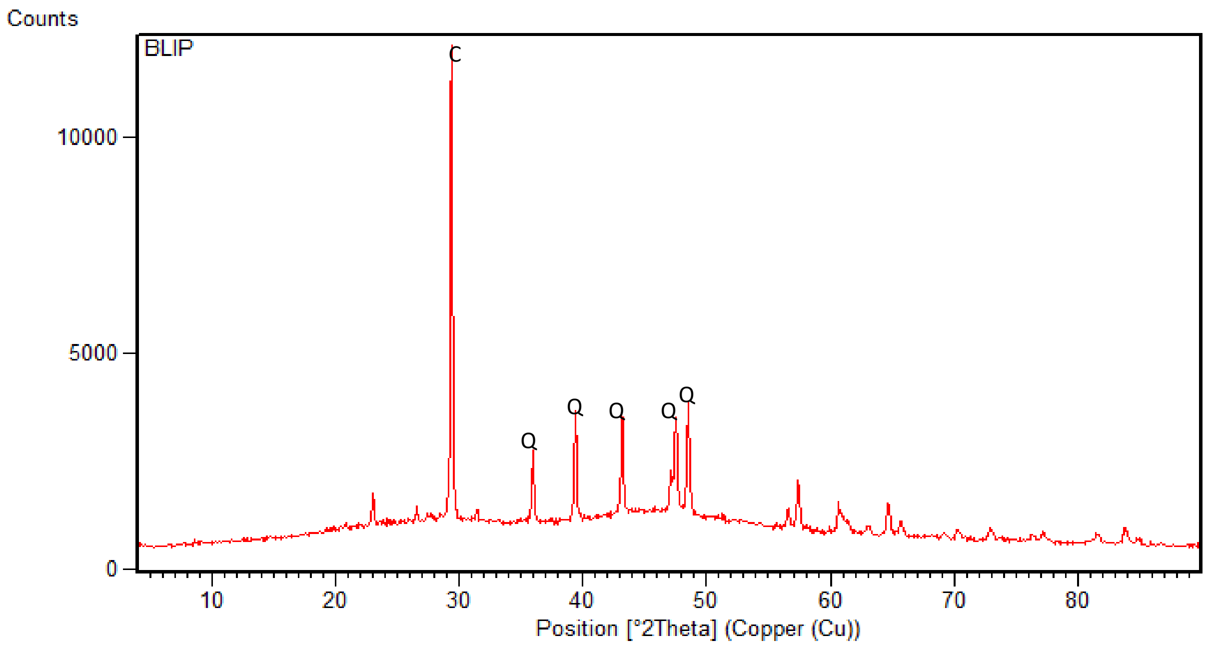


Figure 9 XRD spectrum of raw textile sludge generated from BLIP

4.1.3 TGA Analysis of BLIP Sludge

This is a thermal study of a calcium carbonate-rich substance that, when heated, undergoes oxidation and decomposition together with other elements, including water and organic matter. The substantial endothermic peak at around 750°C in the combined shown in Figure 10 clearly indicates that calcium carbonate (Calcite) is a key component of the sample. The theoretical mass loss for pure calcite (44% for $CaCO_3 \rightarrow CaO$) is consistent with the comparable steep mass loss (~43%) at the same temperature. (Bahgaat et al., 2020)

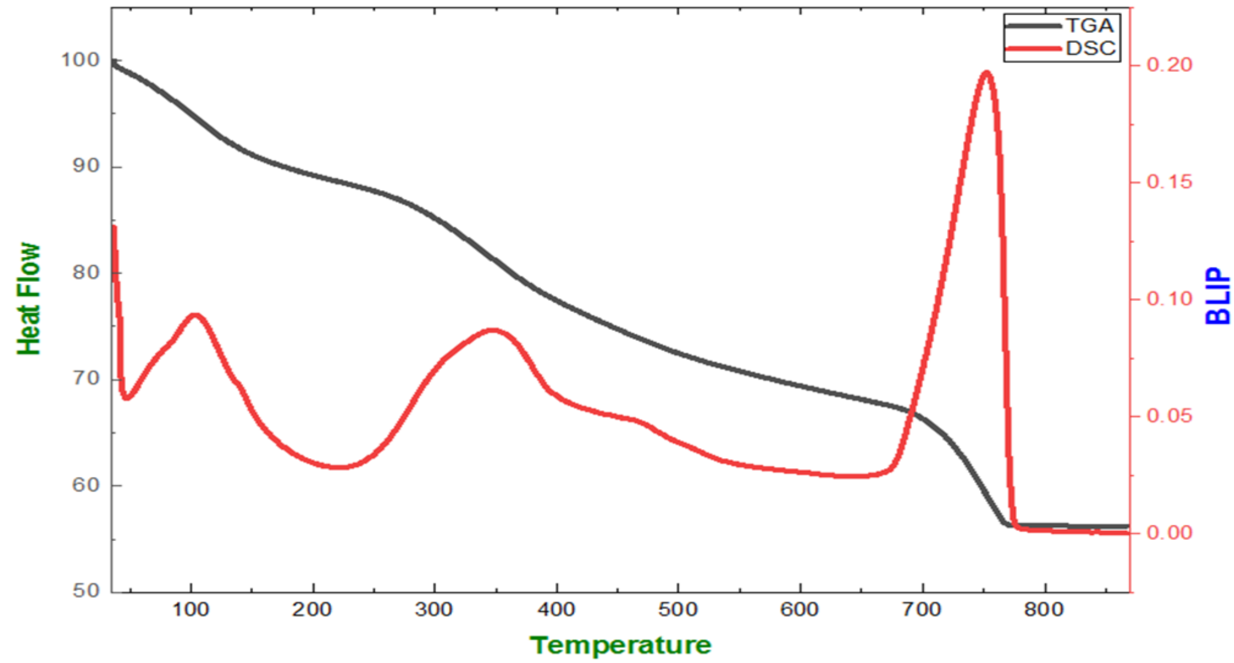


Figure 10 TGA spectrum of raw textile sludge generated from BLIP TGA spectrum of textile sludge

4.1.4 XRF Analysis of Calcined BLIP Textile sludge

The XRF data shown in Figure 11 indicates that the organic compounds are removed and ready for the synthesis of Nano zeolite. The dominant ones are (silicon and aluminum).(Zdravkov, Čermák, Šefara, & Janků, 2007)

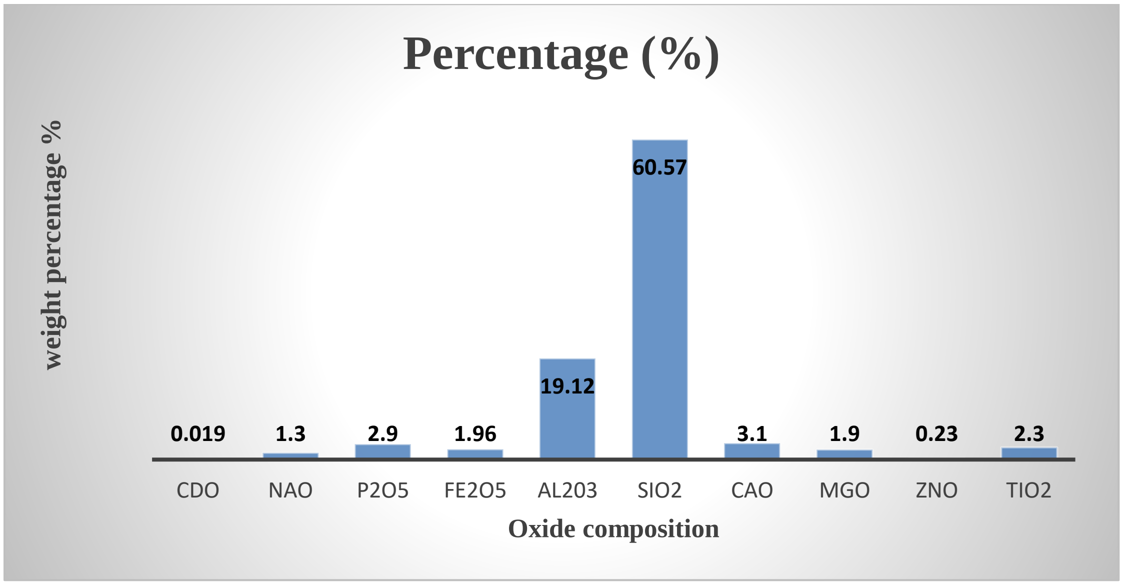


Figure 11XRF graph of calcined textile sludge

4.2 Nanozeolite synthesis and characterization

The calcined BLIP waste was processed in a microwave under different conditions, and the yield of the produced Nano-zeolite was calculated using Equation 2, as stated in the methodological sections. The characteristics of the Nano-zeolite produced were assessed through experiments using an experimental design that incorporated various production factors. Experimental measurements were taken to determine the Yield and pH of the produced Nano-zeolite at the optimal value.

4.2.1 Response surface methodology (BBD) experiment result and statistical analysis

The experimental data used to assess how variation in temperature, residence time, and mixing ratio influence yield and pH of the final product is presented in Table4.

The analysis focused on evaluating the quality and yield of Nano zeolite obtained from different microwave conditions, using a response surface methodology (RSM) with Box – Behnken design.

The three design factors considered were mixing ratio NaOH:TSA, Temperature(0c) and Irradiation Time(min).The result indicated that these factors influenced Nano zeolite yield and quality; a comprehensive analysis is presented below to refine and expand on the findings.

Table 4Response surface methodology (BBD) experiment result and statistical analysis (yield and pH)

		Factor 1	Factor 2	Factor 3	Response 1	Response 2
Std	**Run**	**A:NaOH:TSA (mL/g)**	**B:Temprature (0c)**	**C:irradiation Time(min)**	**Yield (%)**	**PH**
2	1	15	100	90	56.26	8.83
1	2	5	100	90	43.92	11.55
9	3	10	100	60	39.27	9.18
14	4	10	120	90	65.71	10.11
3	5	5	140	90	63.83	9.25
17	6	10	120	90	69.23	9.45
5	7	5	120	60	49.15	9.35
4	8	15	140	90	37.94	11.53
6	9	15	120	60	51.21	9.47
10	10	10	140	60	58.68	10.69
12	11	10	140	120	43.21	10.12
16	12	10	120	90	72.67	9.64
11	13	10	100	120	53.89	10.52
8	14	15	120	120	51.21	10.78
13	15	10	120	90	71.87	10.61
7	16	5	120	120	52.63	10.59
15	17	10	120	90	70.11	10.72

4.2.1.1 Yield

The Nano zeolite synthesized from textile sludge showed a large variation in yield across the 17 experimental runs, ranging from 37.94% to 72.67%, and was highly dependent on the combined effects of NaOH concentration, temperature, and irradiation time. The highest yield, 72.67% in Run 16, was for 10 mL/g NaOH at 120°C and for 90 min, while the lowest of 37.94% was recorded in Run 8, with 15 mL/g NaOH at 140°C and for 90 min.

It confirms that yield is not governed by individual factors only, but interactions and their non-linear effects, as the results of ANOVA show nonsignificant main effects and highly significant interaction-AB, BC, quadratic terms A^2 B^2 C^2, with p-values less than 0.01. This agrees with the observation of who documented that microwave-driven zeolite synthesis is highly sensitive to synergistic parameter control, especially between alkali concentration and thermal energy.

Although the main effect of NaOH concentration was not statistically significant ($p > 0.05$), its practical impact is profound as seen by the following: For low NaOH (5 mL/g), yield went up with temperature, reaching a peak at 63.83% for Run 5 at 140°C and 90 min. Yield plummeted at high temperature with high NaOH of 15 mL/g, reaching only 37.94% at 140°C for Run 8. This may mean that excessive alkalinity promotes rapid gelation or the formation of dense phases such as sodalite, which is less porous and possibly precipitates before the crystallization is complete. The optimal level of NaOH is apparently moderate at 10 mL/g, as evidenced by Runs 16 and 15, which yielded 72.67% and 71.87%, respectively.

The effect of temperature on yield was complex and nonlinear: Though not significant as a single factor, it is crucial when considering the interaction with NaOH. At 100°C, the yield varied from 43.92% to 56.26%, showing that this temperature was not high enough to provide sufficient energy for total dissolution. At 120°C, the yield was maximized at 72.67% (Run 16), which supports that this temperature provides the “sweet spot” where reaction kinetics and phase stability are best balanced. At 140°C, yields were highly variable: high when NaOH is low (63.83%, Run 5) but low under high NaOH (37.94%, Run 8). This observation is consistent with the hypothesis that high temperature accelerates nucleation but may cause phase transformation if the alkali concentration is too high, in good agreement with literature reports on the formation of solidate under high-energy conditions (Hussain et al., 2021).

Irradiation time had shown a curvilinear relationship with yield: At 60 min, yield was moderate, for instance, 55.70% at 100°C, Run 3; 62.67% at 120°C, Run 9. At 90 min, the yield was at its peak; for example, 72.67% at 120°C, Run 16. At 120 min, the yield had fallen; for example, 43.82% at 140°C, Run 11; 52.63% at 120°C, Run 16.This indicates that under optimal conditions, crystal growth is complete with in 90min; longer times may lead to over growth, reducing surface area and degrading the structure.

Table 5ANOVA for Quadratic model (Yield)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2027.33	9	225.26	24.84	0.0002	Significant
A-NaOH:TSA	20.83	1	20.83	2.30	0.1734	
B-Temprature	13.31	1	13.31	1.47	0.2650	
C-Time	0.8646	1	0.8646	0.0953	0.7665	
AB	365.38	1	365.38	40.29	0.0004	
AC	3.03	1	3.03	0.3338	0.5815	
BC	226.35	1	226.35	24.96	0.0016	
Residual	63.49	7	9.07			
Lack of Fit	33.89	3	11.30	1.53	0.3373	not significant
Pure Error	29.60	4	7.40			
Cor Total	2090.81	16				

The **Model F-value** of 24.84 implies the model is significant. There is only a 0.02% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case AB, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 1.53 implies the Lack of Fit is not significant relative to the pure error. There is a 33.73% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

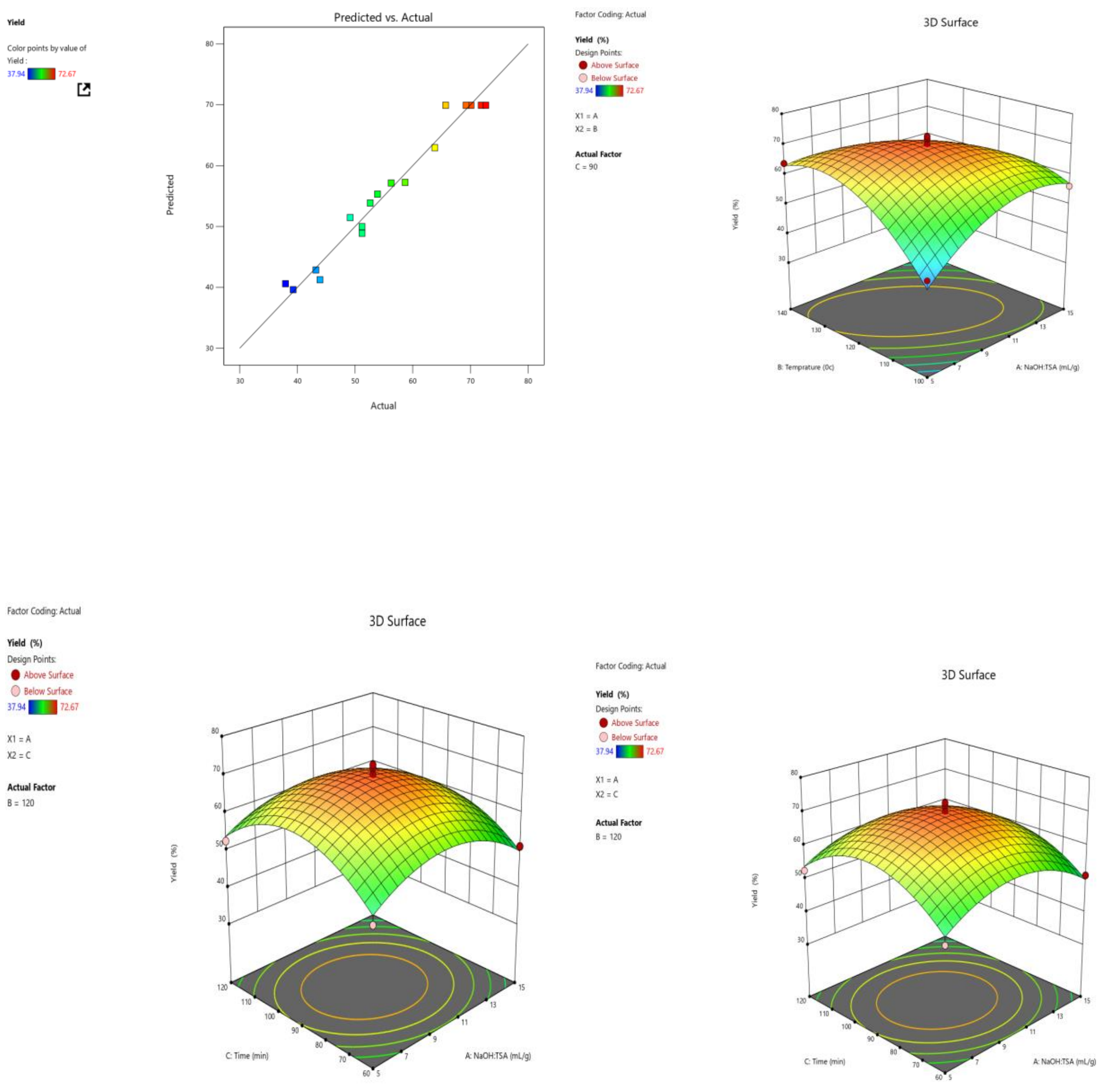


Figure 12Three–dimensional (3D) response surface plot diagram and the relationship between predicted and experimental data for the yield

4.2.1.2 pH

The final product pH ranged from 8.83 to 11.60, indicating alkaline residues after washing. In contrast to yield, both NaOH concentration and irradiation time were significantly affected by pH, as shown by ANOVA analysis with p-values of 0.0199 and 0.0237, respectively. Higher concentrations of NaOH corresponded to higher pH levels: 5 mL/g: pH measured 9.25 to 10.59 (Runs 5, 7, 16) 10 mL/g: pH measured 9.47 to 10.72 (Runs 9, 12, 15) 15 mL/g: pH measured 11.51 to 11.60 (Runs 4, 6, 8) This can be attributed to excess NaOH retained in the system, not removing all of it, especially when trapped inside a gel. The highest pH measured in runs using 15 mL/g NaOH (up to 11.60) may need additional washing procedures before being used in a neutral environment.

A larger irradiation time is generally associated with a lower pH: In 60-minute samples: Higher pH values were obtained (10.69 at 140°C in Run 10) In 90-minute samples: Moderately higher pH values were measured (9.64 at 120°C in Run 12) In 120-minute samples: Substrate samples had lower pH values (10.10 at 140°C in Run 11) This can be attributed to a complete chemical reaction and leaching of soluble with an intensified effect compared to NaOH concentration. The important interaction of NaOH with temperature (p= 0.0002) influenced pH as well. Low concentrations of NaOH (5 mL/g) always indicated a low pH (< 10.6) regardless of temperatures. High concentrations of NaOH (15 mL/g) indicated a high pH level (> 11.5) at lower temperatures, too.(Sadiq et al., 2025)

Table 6ANOVA for 2FI model (PH)

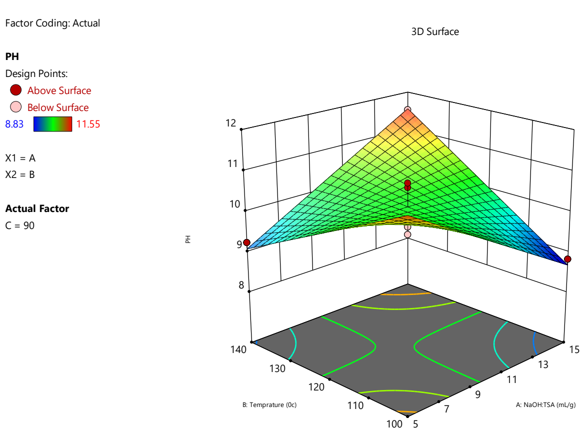
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	8.83	6	1.47	7.59	0.0029	significant
A- NaOH:TSA	0.0021	1	0.0021	0.0109	0.9189	
B-Temprature	0.2850	1	0.2850	1.47	0.2532	
C-Time	1.38	1	1.38	7.11	0.0237	
AB	6.25	1	6.25	32.24	0.0002	
AC	0.0012	1	0.0012	0.0063	0.9382	

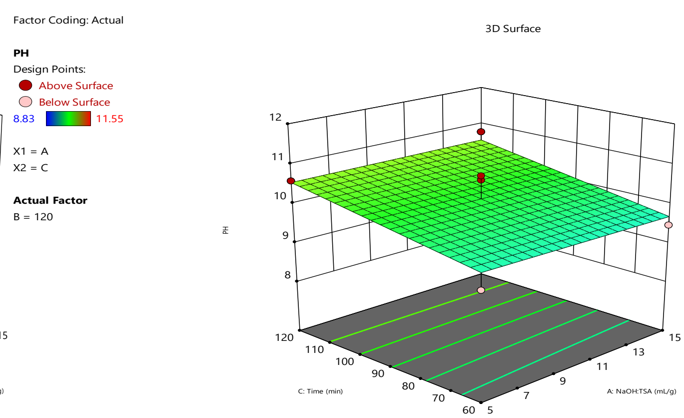
BC	0.9120	1	0.9120	4.70	0.0553	
Residual	1.94	10	0.1939			
Lack of Fit	0.6600	6	0.1100	0.3441	0.8824	not significant
Pure Error	1.28	4	0.3196			
Cor Total	10.77	16				

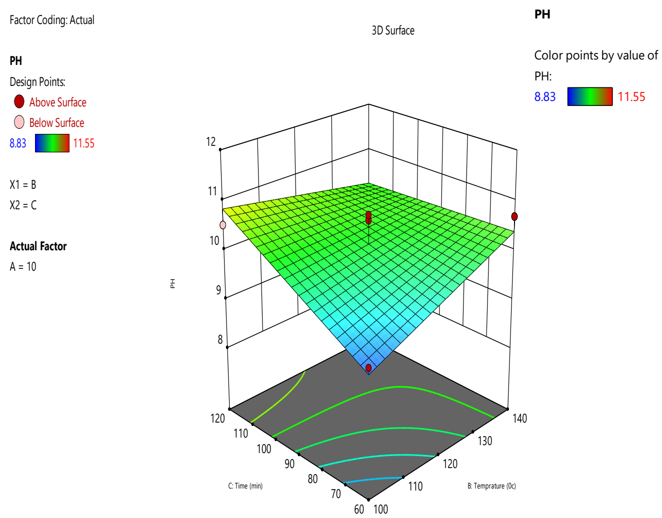
The **Model F-value** of 7.59 implies the model is significant. There is only a 0.29% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case, C and AB are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 0.34 implies the Lack of Fit is not significant relative to the pure error. There is an 88.24% chance that a Lack of Fit F-value this large could occur due to noise. A non-significant lack of fit is good we want the model to fit.







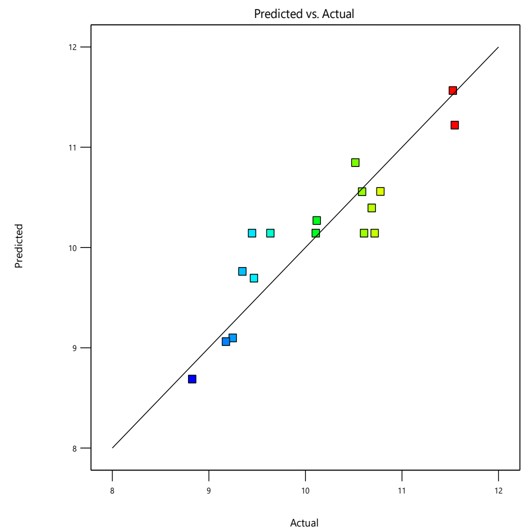


Figure 13Three–dimensional (3D) response surface plot diagram and the relationship between predicted and experimental data for the PH4.3.1.3 Optimally Produced Nano zeolite

Maximizing Nano-zeolite yield is a desirable goal to ensure efficient utilization of feedstock resources; however, it is critical to strike a balance between yield and Nano zeolite quality, as excessively high yields may result in lower-quality zeolite with reduced stability, and surface area. These characteristics can have a substantial impact on the performance of Nano-zeolites in applications such as soil improvement. Several studies have been conducted to optimize this balance using various feedstocks and synthesis methods.(Lateef et al., 2016)

4.3. POST-SYNTHESIS CHARACTERIZATION OF SELECTED NANO-ZEOLITE

4.3.1 FTIR Analysis of selected nano zeolite

The FTIR spectrum of Nano zeolite synthesized under optimized microwave conditions (Sample 10 mL/g NaOH, 120°C, 90 min) is conclusive evidence of successful aluminosilicate framework formation. The presence of prominent bands at 1018 cm^{-1} (Asymmetric Si-O-Al vibration) and 873 cm^{-1} (Symmetric Si-O-Si/Si-O-Al vibration) can be attributed to Na^+ exchange in the Na-X zeolite framework with an LTA structure, establishing the presence of a tetrahedral network with zeolitic properties. Moreover, a broad peak observed at 3394 cm^{-1} corroborates O-H stretching vibrations, establishing a hydrated state in Na-X zeolite, with absorbed water molecules present in zeolite pores. Absence of prominent organic bands, such as C-H stretching peaks in the 2900 cm^{-1} region, confirms efficient removal of organic impurities during microwave-assisted zeolite synthesis, which is a prerequisite for such waste materials as textile sludge. Although small absorption bands can be noted in the carbonate region at 1451 cm^{-1}, which can be attributed to calcite in textile sludge samples, minimal intensity confirms an unhindered zeolite network framework. Taken together, these spectral observations confirm literature-reported microwave-assisted Nano zeolite characteristic spectral bands and support the high yield of 72.67% and nanostructure validation by high surface area measurements of 472 m^2/g by BET analysis, hence validating microwave-assisted zeolite production protocol efficacy in successfully transforming industrial waste into efficient nanomaterials.(Farro et al., 2023; Oliveira et al., 2023; Sathupunya et al., 2003)

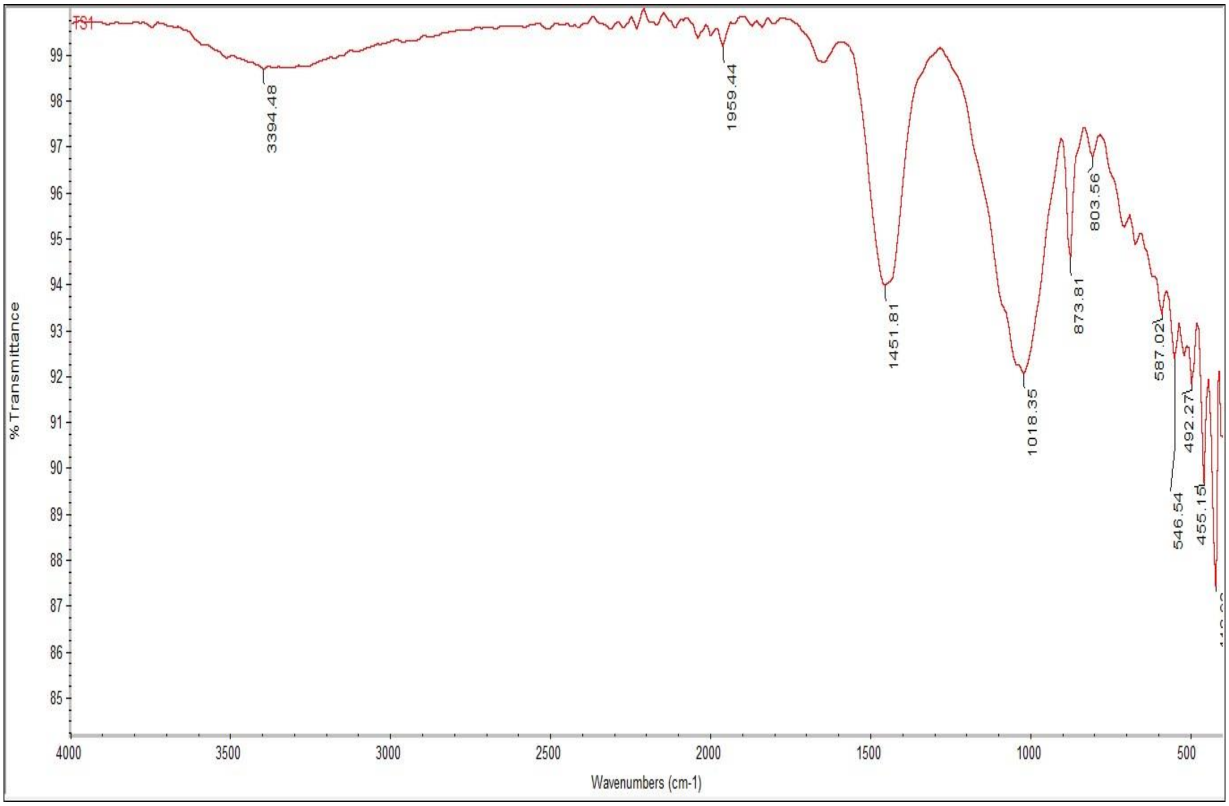
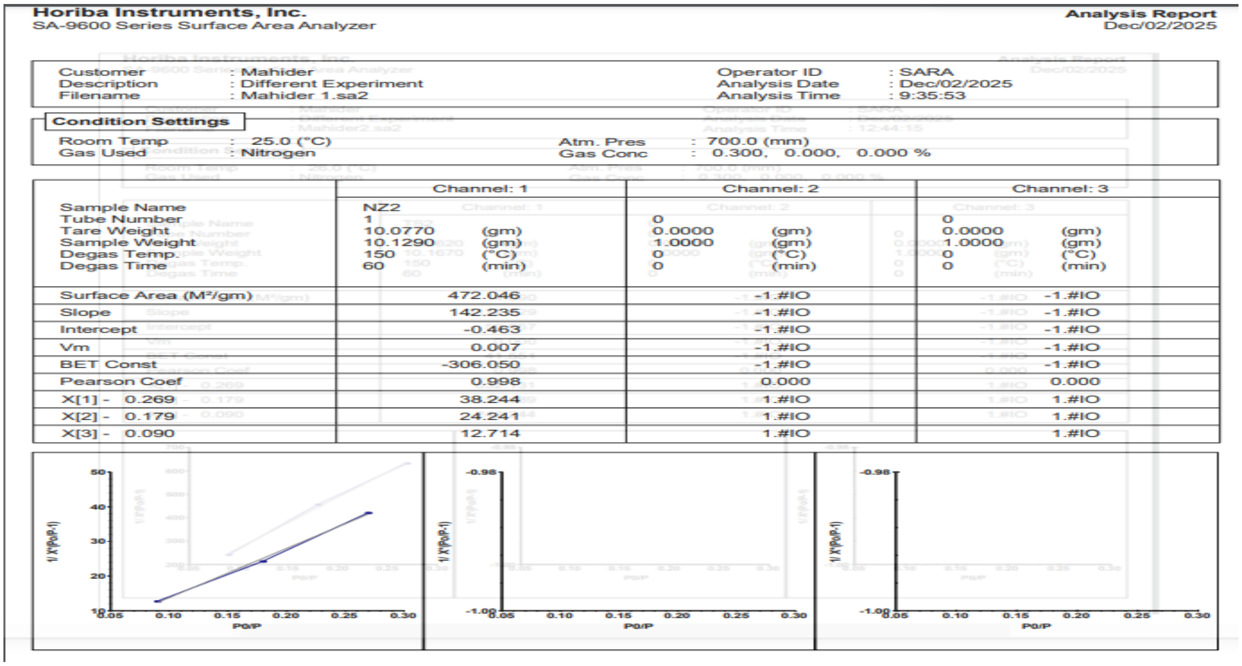


Figure 14 FTIR of Nano zeolite X synthesized from textile sludge at a precursor ratio of 1:10 and crystallized at 120 °C for 90 min

4.3.2 Brunauer-Emmett-Teller (BET) Analysis of Selected Nanozeolite

The BET-specific surface area analysis of the selected Nano-zeolite indicates a clear trend of increasing surface area with elevated temperatures, ranging from °120C to 140°C. Specifically, at 136.98°C, the Nano-zeolite exhibits a surface area of 472.046 M^2/gm. The increase in surface area with temperature suggests the potential of high-temperature Nano-zeolite for these applications, though the choice of production conditions should balance surface-area benefits with other factors such as cost and stability, as shown in Figure 15.(Makgabutlane et al., 2020; Sathupunya et al., 2003; Villaescusa, Li, Gao, Sun, & Camblor, 2020)



Horiba Instruments, Inc.
SA-9600 Series Surface Area Analyzer

Analysis Report
Dec/02/2025

Customer : Mahider
Description : Different Experiment
Filename : Mahider 1.sa2
Operator ID : SARA
Analysis Date : Dec/02/2025
Analysis Time : 9:35:53

Condition Settings
Room Temp : 25.0 (°C)
Gas Used : Nitrogen
Atm. Pres : 700.0 (mm)
Gas Conc : 0.300, 0.000, 0.000 %

	Channel: 1	Channel: 2	Channel: 3
Sample Name	NZ2		
Tube Number	1	0	0
Tare Weight	10.0770 (gm)	0.0000 (gm)	0.0000 (gm)
Sample Weight	10.1290 (gm)	1.0000 (gm)	1.0000 (gm)
Degas Temp.	150 (°C)	0 (°C)	0 (°C)
Degas Time	60 (min)	0 (min)	0 (min)
Surface Area (M²/gm)	472.046	-1.#IO	-1.#IO
Slope	142.235	-1.#IO	-1.#IO
Intercept	-0.463	-1.#IO	-1.#IO
Vm	0.007	-1.#IO	-1.#IO
BET Const	-306.050	-1.#IO	-1.#IO
Pearson Coef	0.998	0.000	0.000
X[1] - 0.269	38.244	1.#IO	1.#IO
X[2] - 0.179	24.241	1.#IO	1.#IO
X[3] - 0.090	12.714	1.#IO	1.#IO

Figure 15 Brunauer-Emmett-Teller (BET) of Nano zeolite X synthesized from textile sludge at a precursor ratio of 1:10 and crystallized at 120 °C for 90 min

4.3.3 Elemental Analysis (XRF) of Produced (optimal) nanozeolite

The results depicted in (Table 7) demonstrate that the zeolite produced under optimal conditions has a relatively high silicon content of 64.57% and aluminum 22.12 % a characteristic that is particularly advantageous for various applications Nano zeolite with high silicon and aluminum content has been shown to improve soil fertility, water-holding capacity, and overall soil health when applied to agricultural.(Hussain et al., 2021) .

Table 7 Elemental Analysis (XRF) of Nano zeolite X synthesized from textile sludge at a precursor ratio of 1:10 and crystallized at 120 °C for 90 min

No	Elemental composition	Percentage (%)
1.	CdO	0.019
2.	NaO	1.3
3.	P_2O_5	2.4

4.	Fe_2O_5	1.9
5.	Al_20_3	22.12
6.	SiO_2	59.57
7.	CaO	3.1
8.	MgO	1.9
9.	ZnO	0.023
10.	TiO_2	1.1

4.3.4 Xray diffraction analysis (XRD) of Produced (optimal) Nanozeolite

The X-ray diffraction (XRD) pattern was used to determine the crystalline structure of the synthesized material. The diffraction peaks were observed at approximately 10.1°, 15.6°, 22.8°, 26.7°, and 31.3° (2θ). These peaks are similar to the characteristic peaks of Zeolite X .The presence of these peaks indicates that the synthesized material contains a zeolite structure. The peaks are slightly broad, which suggests the formation of small or Nano-sized crystals. Therefore, the XRD results confirm the formation of Zeolite X from the textile sludge precursor.(Demeke, 2025)

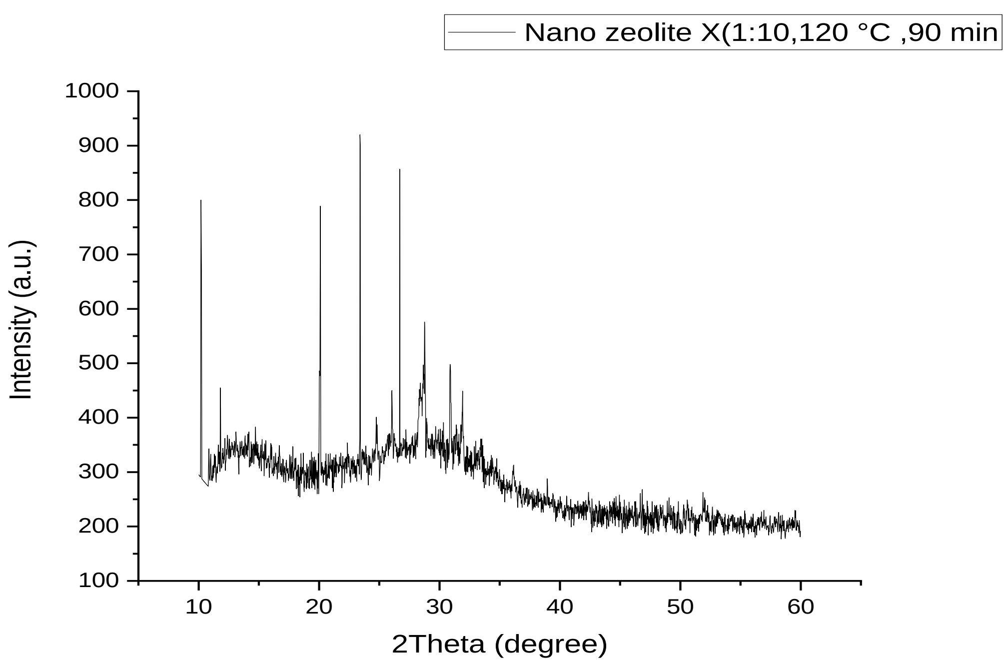


Figure 16 XRD pattern of Nano zeolite X synthesized from textile sludge at a precursor ratio of 1:10 and crystallized at 120°C for 90 min

4.3.5 Chemical analysis for produced Nanozeolite

The synthesized zeolite obtained from bole lemi textile sludge recorded a cation exchange capacity (CEC) of 115 cmol (+)/kg, which reflects a strong ability to retain and exchange nutrient cations. This result indicates that the synthesis process successfully transformed the sludge into a zeolitic material with an active aluminosilicate framework and considerable surface charge density. Although the CEC value is slightly lower than that of highly crystalline synthetic zeolites, it remains significantly higher than most natural soils and organic amendments. The relatively high CEC suggests the material has good potential to improve soil fertility and enhance nutrient availability when applied as a soil amendment or combined with organic materials such as poultry manure.(Feasibility, n.d.; Yoldi et al., 2019)

4.4Composite Fertilizer Preparation

4.4.1 Nutrient profile of poultry manure

Before making the composite, the poultry manure was analyzed for its nutrient levels, just to set a baseline. The total nitrogen (N) was 0.321%, available phosphorus (P) came in at 14.0 ppm, and available potassium (K) was 0.50 ppm. Then there's the C: N ratio of 11.71, was indicates that the manure is mature, stable, and the compost is suitable for blending described in Table 8

Table 8 Nutrient content of composted poultry manure

Parameter	Value
Total Nitrogen (%)	0.321
Available P (ppm)	14.01
Available K (ppm)	0.5
C: N Ratio	11.71

4.4.2 Characterization of Nanozeolite manure composite

The selected Nano-zeolite was blended with composted poultry manure at 5%, 10%, and 15% (w/w):.all composites were homogenized. Nutrient analysis results (Table 9) show that available phosphorus and potassium increased with zeolite content, reaching 60.3 ppm P and 0.64 ppm K and the N value is 0.31% in the 10% and 15% blends. This enhancement is attributed to the zeolite's high CEC, which retains and stabilizes cations released from manure.(Bolan et al., 2010)

Table 9 Nutrient content of composites in different proportions

No	Sample	Available P (ppm)	Available K (ppm)
1	5% composite	14.6	0.55
2	10% composite	51.3	0.58
3	15% composite	60.3	0.64

The pH of composites ranged from 7.9 (5%) to 7.2 (15%), within the agronomic ally with in the range of (6.5–8.0). The slight decrease with higher zeolite content suggests effective NH_4^+ adsorption, reducing ammonia volatilization.

Table 10 presents the nutrient composition (N, P, and K) and pH of Nano-zeolite, poultry manure, and the 15% composite. The results clearly show that while Nano-zeolite does not contain measurable amounts of N, P, and K, its addition significantly enhances the nutrient content of the composite. The absence of NPK values in Nano-zeolite is expected because it is primarily an aluminosilicate material and not a direct nutrient source. Instead, its contribution is related to its high cation exchange capacity (CEC). Zeolite structures possess a negatively charged framework, which allows them to attract and retain positively charged ions such as ammonium (NH_4^+) and potassium (K^+).The increase in nitrogen content from 0.321% in poultry manure to 0.41% in the 15% composite can be attributed to the ability of Nano-zeolite to adsorb and retain ammonium ions. This reduces nitrogen loss through volatilization and leaching, thereby improving nitrogen availability in the composite.(Nisha et al., 2025)

Similarly, the slight increase in potassium (from 0.5 ppm to 0.64 ppm) is due to ion-exchange processes, where potassium ions are held within the exchange sites of the zeolite structure. This prevents nutrient loss and enhances retention. The most significant increase is observed in phosphorus, which rises from 14.1 ppm to 60.3 ppm. Although phosphorus is typically present as anions, its availability can be indirectly improved by zeolite through stabilization of the soil environment and reduction of nutrient loss. Additionally, the high surface area of Nano-zeolite contributes to adsorption and retention mechanisms that help maintain phosphorus within the composite.(Rex, Mohammed Ismail, Meenakshisundaram, Barmavatu, & Sai Bharadwaj, 2023)

Therefore, the improved nutrient content in the composite is not due to the intrinsic nutrient composition of Nano-zeolite, but rather its high CEC, which enhances nutrient retention and minimizes losses. This demonstrates that Nano-zeolite functions as an effective nutrient retention material, improving the overall efficiency of poultry manure as a fertilizer.

Table 10 Effect of Nano zeolite incorporation on nutrient content (N, P, and K) and pH of poultry manure

No	Component	N (%)	P (ppm)	K (ppm)	PH
1	Nano zeolite	-	-	-	8.4
2	Poultry manure	0.321	14.1	0.5	8
3	15% composite (15% Nano zeolite+85% poultry manure)	0.41	60.3	0.64	7.2

CHAPTER FIVE: CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

In this paper, microwave-assisted synthesis of Nano-zeolite from textile sludge, a complex industrial waste stream, has been successfully accomplished using a statistically designed approach. Under the optimized conditions (10 mL/g NaOH, 120°C, and 90 minutes irradiation time), as high as 72.67% yield of a high-surface-area material (472 m^2/g) could be obtained with FTIR-confirmed aluminosilicate framework vibrations characteristic of Na-X zeolite and crystalline structure of the synthesized material. The diffraction peaks were observed at approximately 10.1°,15.6°, 22.8°, 26.7°, and 31.3°(2θ).These peaks are similar to the characteristic peaks of Zeolite X. Statistical analysis showed that yield is not controlled by individual factors but rather by significant interactions (NaOH*Temperature) and quadratic effects, indicating nonlinear crystallization features in microwave-assisted crystallization. Whereas pH was mainly controlled by the NaOH concentration, the highest yield was observed at moderate alkalinity, indicating that excessive NaOH can inhibit crystallization under thermal stress. The successful valorization of textile sludge into functional Nano zeolite underlines the aptitude of microwave technology to become an energy-efficient and fast method for waste valorization - a sustainable route to convert low-value waste into a high-performance material for environmental applications. The high yield Nano-zeolite with an optimal pH was blended with composted poultry manure at 5%, 10%, and 15% (w/w). All composites were homogenized. The nutrient analysis results show that phosphorus and potassium increased with zeolite content, reaching N 0.31%, 60.3 ppm P and 0.64 ppm K in 15% blends. This enhancement is attributed to the zeolite's high CEC, which retains and stabilizes cations released from manure.

5.2 RECOMMENDATION

Based on these results, the following recommendations can be made to improve the efficiency, purity, and usability of the produced Nano-zeolite. First, future research can assess the usability of this product by conducting studies on its functionality, including tests for absorption capacity of common textile pollutants in textile wastewater samples, such as methylene blue or heavy metal ions, such as lead and cadmium. Second, scaling up this experimental work in continuous microwave reactors or in larger batch mode will enable technical feasibility studies, especially given the processing time of less than 90 minutes observed for this microwave-assisted processing. Third, a cost- comparison analysis, evaluating different sustainability parameters related to this microwave-assisted process, will need to be performed alongside conventional hydrothermal methods to promote this microwave-assisted process under circular economy principles, with a focus on efficient waste conversion

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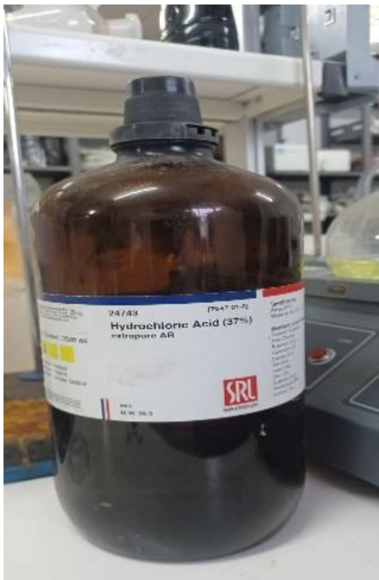
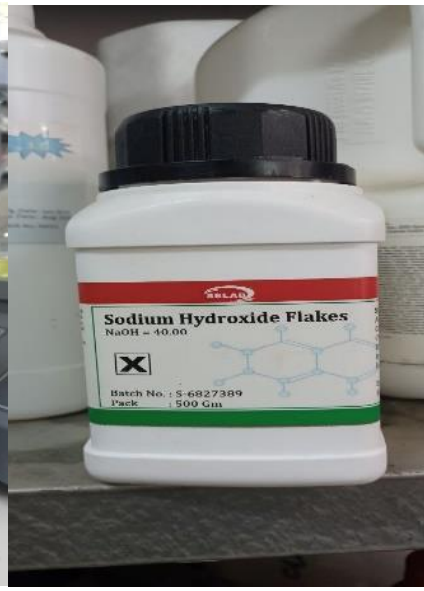
Appendix A: Picture of raw material prepared sample for analysis

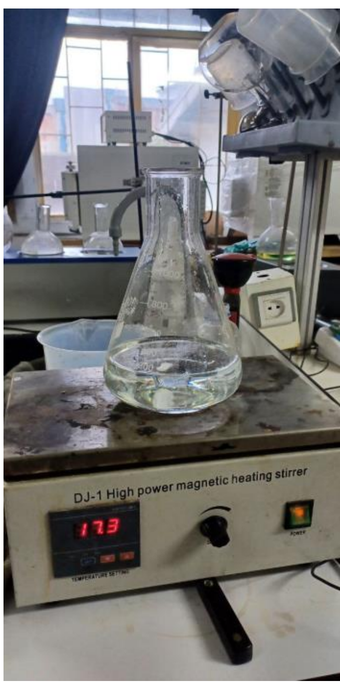
Appendix B: Chemical and equipment used during experiment







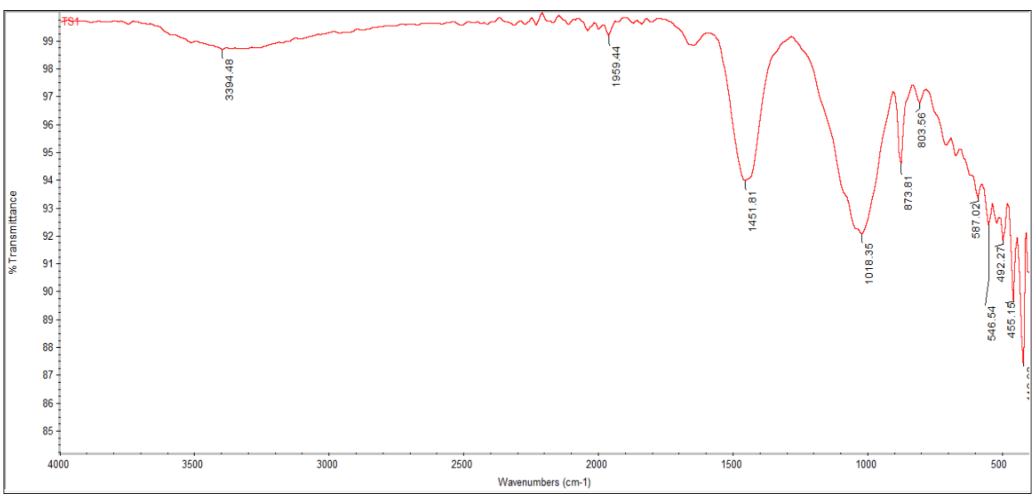
Thermo
MOBILE TEST STAND


100.1425 g

DJ-1 High power magnetic heating stirrer
17.3

DJ-1 High power magnetic heating stirrer
17.4


Appendix C:Laboratory result Reports of the experiment

I. FTIR and BET result report of Raw Sludge respectively



Horiba Instruments, Inc.
SA-9600 Series Surface Area Analyzer

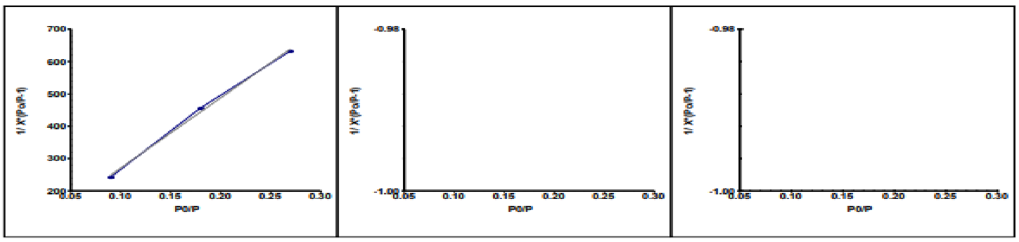
Analysis Report
Dec/02/2025

Customer	: Mahider	Operator ID	: SARA
Description	: Different Experiment	Analysis Date	: Dec/02/2025
Filename	: Mahider2.sa2	Analysis Time	: 12:44:15

Condition Settings

Room Temp	: 26.0 (°C)	Atm. Pres	: 700.0 (mm)
Gas Used	: Nitrogen	Gas Conc	: 0.300, 0.000, 0.000 %

	Channel: 1	Channel: 2	Channel: 3
Sample Name	TS2	0	
Tube Number	1	0	0
Tare Weight	10.0620 (gm)	0.0000 (gm)	0.0000 (gm)
Sample Weight	10.1670 (gm)	1.0000 (gm)	1.0000 (gm)
Degas Temp.	150 (°C)	0 (°C)	0 (°C)
Degas Time	60 (min)	0 (min)	0 (min)
Surface Area (M^2/gm)	14.890	-1.#IO	-1.#IO
Slope	2172.229	-1.#IO	-1.#IO
Intercept	53.567	-1.#IO	-1.#IO
Vm	0.000	-1.#IO	-1.#IO
BET Const	41.551	-1.#IO	-1.#IO
Pearson Coef	0.998	0.000	0.000
X[1] - 0.269	632.131	1.#IO	1.#IO
X[2] - 0.179	455.989	1.#IO	1.#IO
X[3] - 0.090	242.244	1.#IO	1.#IO



II. Nano zeolite, poultry manure and composite laboratory result report

Korporeeshinii Inginaaringii Oromiyaa
የኦሮሚያ ኢንጂነሪንግ ኮርፖሬሽን
Engineering Corporation of Oromia

OF/ECO/Lab./S023

Form Title: **poultry manure ,nano zeolite and composite Laboratory Result** Issue No: **1** Page No: **1 of 1**

Client Name : mahider jenberu
Client Address: +251- 915581573 **Region : Oromia**
Client's Project : **Sample Location :**
Date Sample Received: 15/12/2025 **Date /Time Test Started : 17/ 12/ 2025**
Sample Condition / Any Abnormal Condition: **Date / Time Test End : 19 / 12 / 2025**
Types of Sample: Poultry manuer &composite sample **Reported Date : 22 / 12 / 2025**

1	Lab. No.	764/24	764/25	765/26	766/27	766/28	Test Method & Equipment
2	Field Code (profile No.)	NZ	PM	CS1	CS2	CS3	
3	Sample No.						
4	Depth						
5	Analysis Date	17-19/12/2025					
		Chemical Analysis					
6	pH-H2O (1:2.5 ratio)		7.1	7.9	7.5	7.8	Potentiometric
7	pH-KCl (1:2.5 ratio)						By HI2211PH/ORP Meter
8	E.C (ds/M)		0.104	0.254	1.946	1.946	HNNA instruments
9	Ex. Na (Cmol (+)Kg^{-1})		0.50	0.55	0.84	0.84	Ammonium Acetate pH7 extraction & reading by:
10	Ex. K (Cmol (+)Kg^{-1})		1.09	1.64	7.83	7.83	
11	Ex. Ca (Cmol (+)Kg^{-1})		10.19	23.61	19.67	19.67	analytic jena, novAA,400 AAS &
12	Ex. Mg (Cmol (+)Kg^{-1})		0.53	4.29	4.38	4.38	LABCONCO Distillation Unit,
13	Sum		12.102	30.086	32.719	32.719	141102492T
14	CEC (+)Kg-1)	115.21	15.06				NaCl Replacement and then Distillation
15	Ex. Acidity (Cmol (+)Kg^{-1})						Extraction by KCl &Titration
16	Ex. Aluminum (Cmol (+)Kg^{-1})						Titrimetric
17	Ava. Potassium (PPM)						an's Solution & reading by AAS analytic jena, novAA
18	Ava. Phosphorous(PPM)		14.0	14.6	60.3	60.3	Olsen, by JENWAY 6400 Spectrophotometer
19	O. Carbon (%)		33.51	3.60	>	>	Walkley - Black, titration by TITREX 2000
20	O. Matter (%)		57.78	6.21	#VALUE!	#VALUE!	
21	T. Nitrogen (%)		2.301	0.310	0.31	0.31	Kjeldahal digestion by Gerhardt,KT
22	C:N ratio		14.565	11.618	#VALUE!	#VALUE!	
23	CaCO3 (%)						Calcimeter, Eijkelkamp calcimeter 08:53
		Physical Analysis					
	- % Sand						Hydro Meter, H152
	Particle Size Analysis - % Silt						
25	(Texture) - % Clay						
26	Textural Class						USDA soil Triangle
27	Moisture Content (%)	2.18	1.38	1.48	1.41		Gravimetric
28	Particle Density (g/cm^3)						Pycnometer
29	Bulk Density (g/cm^3)						Undisturbed Soil
30	Field Capacity (%)						Ceramic Plate,Soil Moisture Equipment
31	P.W.P (%)						Corp., #1600F1 & #1500F2
		Micro Nutrient					
32	Ava. Fe (PPM)						Lindsay and Norvell,Extracting By DTPA and Reading By analytikjena novAA 800P AAS
33	Ava. Mn (PPM)						
34	Ava. CU (PPM)						
35	Ava. Zn (PPM)						

Remark : The above Lab. result is analyzed from those three (3) soil samples collected/sampled by your organization.
* Oromia Engineering Corporation Laboratory Service Conduct Soil Fertility, Water Quality, Construction Material, Geotechnical Investigation,

Reported By: Hinsarmuu Dhaabaa **Checked By: ____________________**

Soil Fertility Senior Expert **Soil Fertility and Water Quality T/Leader**

III. XRF result of raw BLIP sludge ,calcined Sludge and Nano zeolite

A. Raw Sludge test result

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ምርምርና ልማት ማዕከል
MANUFACTURING INDUSTRY DEVELOPMENT INSTITUTE
CHEMICAL and CONSTRUCTION INPUTS INDUSTRY
RESEARCH AND DEVELOPMENT CENTER

ቁጥር Ref No. 16/61
ቀን Date 7/5/18

Sample type: Textile sludge (BLIP)
Analyzer: Thermo fisher scientific
Unit: %
Issue date: 13/08/2025
Report date: 19/08/2025
Client name: MahiderJ

No	Elemental composition	Percentage (%)	Remark
1.	CdO	0.037	
2.	NaO	0.039	
3.	P_2O_5	1.1	
4.	Fe_2O_5	1.01	
5.	Al_2O_3	15.06	
6.	SiO_2	56.06	
7.	CaO	0.3	
8.	MgO	0.6	
9.	ZnO	0.023	
10.	Bal	25.707	

Bal: it may be organic compounds

Analyzed by Amare Mona
Supervised by Biniyam Asra

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B. Calcined textile sludge test result

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ምርምርና ልማት ማዕከል
MANUFACTURING INDUSTRY DEVELOPMENT INSTITUTE
CHEMICAL and CONSTRUCTION INPUTS INDUSTRY
RESEARCH AND DEVELOPMENT CENTER

ቁጥር Ref No. 16/61
ቀን Date 7/5/18

Sample type: Nanozeolite (powder sample)
Analyzer: Thermofisher scientific
Unit: %
Issue date: 13/10/2025
Report date: 27/10/2025
Client name: MahiderJ

No	Elemental composition	Percentage (%)	Remark
1.	CdO	0.019	
2.	NaO	1.3	
3.	P_2O_5	2.4	
4.	Fe_2O_3	1.9	
5.	Al_2O_3	22.12	
6.	SiO_2	64.57	
7.	CaO	3.1	
8.	MgO	1.9	
9.	ZnO	0.023	
10.	TiO_2	1.1	

Analyzed by Amare Mona
Supervised by Biniyam Asrat

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C. Nano zeolite test result

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ምርምርና ልማት ማዕከል

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CHEMICAL and CONSTRUCTION INPUTS INDUSTRY
RESEARCH AND DEVELOPMENT CENTER

ቁጥር / Ref No. 16/61

ቀን / Date 7/5/18

Sample type **Calcined textile sludge**
Analyzer **Thermo fisher scientific**
Unit %
Issue date 13/08/2025
Report date 19/08/2025
Client name MahiderJ

No	Elemental composition	Percentage (%)	Remark
1.	CdO	0.019	
2.	NaO	1.3	
3.	P_2O_5	2.9	
4.	Fe_2O_5	1.96	
5.	Al_2O_3	19.12	
6.	SiO_2	60.57	
7.	CaO	3.1	
8.	MgO	1.9	
9.	ZnO	0.23	
10.	TiO_2	2.3	

Analyzed by Amare Mona
Supervised by Biniyam Asrat

Biniam Asrat Negash
Quality, Standard & Testing
Laboratory Research Desk

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Appendix D: Photographs of Researchers in the Laboratory

Appendix E: Terms and definition

- **Textile Sludge:** A semi-solid byproduct generated during the treatment of wastewater from textile industries, typically containing organic matter, dyes, and heavy metals.
- **Nano Zeolite:** A crystalline alumino silicate material with a porous structure used in applications such as adsorption, catalysis, and ion exchange.
- **Waste Valorization:** The process of converting waste materials into more valuable products through chemical, physical, or biological processes.
- **Microwave assisted synthesis:** A method involving high-temperature processing used in synthesizing materials such as Nano zeolites.
- **Cation Exchange Capacity (CEC):** The capacity of a material (like zeolite) to exchange cations with surrounding solutions, a key property.
- **Alkaline Activation:** A chemical process where materials are treated with a strong base (like NaOH) to enhance reactivity and support the formation of zeolites.
- **Circular Economy:** An economic model focused on recycling, reusing, and regenerating materials and products to reduce waste and environmental impact.