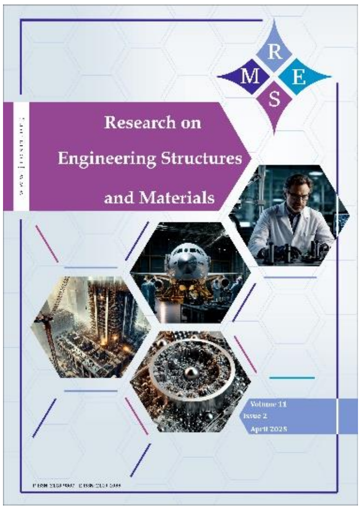
Research on Engineering Structures & Materials
www.jresm.org
Research Group



Effect of waste paper-based nanocellulose synthesized at low temperatures on the microstructural properties of mortar

Saloma, Siti Aisyah Nurjannah, Anis Saggaff, Arie Putra Usman, Pengki Suanto, Rifkah

Online Publication Date: 20 November 2025

URL: http://www.jresm.org/archive/resm2025-1100ma0820rs.html
DOI: http://dx.doi.org/10.17515/resm2025-1100ma0820rs

Journal Abbreviation: *Res. Eng. Struct. Mater.*

To cite this article
Saloma, Nurjannah S A, Saggaff A, Usman A P, Suanto P, Rifkah. Effect of waste paper-based nanocellulose synthesized at low temperatures on the microstructural properties of mortar. *Res. Eng. Struct. Mater.*, 2026; 12(3): 1609-1635.

Research on Engineering Structures & Materials
www.jresm.org
MIM Research Group

Research Article

Effect of waste paper-based nanocellulose synthesized at low temperatures on the microstructural properties of mortar

Saloma [*,1,a], Siti Aisyah Nurjannah [1,b], Anis Saggaff [1,c], Arie Putra Usman [1,d], Pengki Suanto [2,e], Rifkah [3,f]

[1]*Departement of Civil Engineering, Faculty of Engineering, Universitas Sriwijaya, Indonesia*
[2]*Departement of Civil Engineering, Faculty of Science and Technology, Pradita University, Indonesia*
[3]*Engineering Science, Doctoral Program, Faculty of Engineering, Universitas Sriwijaya, Indonesia*

Article Info

Article History:

Received 20 Aug 2025
Accepted 20 Nov 2025

Keywords:

Waste paper-based;
Nanocellulose (NC);
Utilization of waste paper;
Low temperature synthesis;
Mortar sustainability;
Materials sustainability;
Environmentally friendly materials

Abstract

The study explores using nanocellulose synthesized from waste paper as an additive in mortar, focusing on its impact on the material's microstructure. Nanocellulose, derived from waste paper, offers environmental benefits by reducing waste and utilizing low-temperature synthesis methods that decrease energy consumption and carbon emissions. The research evaluates how the addition of nanocellulose influences the microstructural properties of mortar, including porosity, density, pore size distribution, and mechanical strength. The results indicate that incorporating nanocellulose improves mortar's compressive strength, crack resistance, and water penetration resistance. In particular, nanocellulose enhances the bond between cement particles and aggregates, reduces porosity, and increases the material's density, making it more durable and resistant to environmental factors. The study highlights the role of nanocellulose in facilitating better dispersion of cement particles, which results in homogeneous pore size distribution and improved material performance. Furthermore, the research compares the effects of nanocellulose, cellulose fiber, and cellulose ash on mortar properties, revealing that nanocellulose provides superior performance in enhancing mortar's mechanical strength and sustainability. The study's findings contribute to developing eco-friendly, high-performance building materials by promoting waste paper-derived nanocellulose in construction. Using nanocellulose addresses waste management issues and supports the creation of stronger, more durable, environmentally friendly construction materials.

1. Introduction

Waste paper management has become a significant concern in global efforts to achieve sustainability. The increasing amount of waste paper from various sectors, including offices, industries, and households, poses substantial environmental problems if not appropriately managed. One solution that has attracted attention is using waste paper as a raw material to produce nanocellulose. This nanomaterial has excellent potential for application in various fields, including construction. Studies have shown that nanocellulose produced from waste paper can significantly improve the microstructural properties of mortar, which is the primary material in building construction [1]. As an environmentally friendly and sustainable material, nanocellulose has excellent mechanical properties, such as high tensile strength, high elastic modulus, and the ability to form strong hydrogen bonds. These properties make it a potential additive to improve the microstructure of mortar. In recent studies, nanocellulose synthesized from waste paper at low

---

[*]Corresponding author: salomaunsri@gmail.com

[a]orcid.org/0000-0003-4302-0282; [b]orcid.org/0000-0003-3058-592X; [c]orcid.org/0000-0002-3281-2754; [d]orcid.org/0000-0002-1982-6561[e]orcid.org/0000-0001-8546-7870; [f]orcid.org/0009-0003-4182-900X

DOI: http://dx.doi.org/10.17515/resm2025-1100ma0820rs

temperatures showed promising results in improving mortar quality, including compressive strength, resistance to cracking, and dimensional stability [2,3].

Nanocellulose synthesis from waste paper involves hydrolysis, including NaOH and NaClO synthesis, lignin removal, and nanoscale fiber separation [4]. Low temperatures in this process save energy and reduce carbon emissions, making it more sustainable than conventional approaches. In addition, this process produces nanocellulose with a more uniform particle size, which is important for application in mortar. Adding nanocellulose to mortar allows for an even distribution of nanofibers, thereby improving the bond between cement particles and aggregates [5]. From a microstructural perspective, nanocellulose can fill micropores in mortar, reduce porosity, and increase the density of the material. This contributes to increased compressive strength and resistance to water penetration. In addition, nanocellulose's hydrophilic nature helps control the mortar's water content during the setting and hardening process, ultimately improving the mortar's final quality. A study by [6] showed that adding 1% nanocellulose to mortar can increase compressive strength by up to 25% compared to conventional mortar. The success of nanocellulose application in mortar also depends on parameters such as nanocellulose concentration, mixing method, and basic mortar characteristics.

In a study by [7], it was stated that mixing nanocellulose with the help of ultrasonic technology can ensure better distribution and prevent agglomeration of nanoparticles. In addition, this study also showed that the optimal concentration of nanocellulose ranges from 0.5% to 2%, depending on the type of cement and aggregate used. The environmental impact of using nanocellulose in mortar is also noteworthy. Utilizing waste paper as a raw material helps reduce the amount of waste in landfills. In addition, reducing energy consumption during low-temperature nanocellulose synthesis contributes positively to the overall carbon footprint. A study by [8] confirmed that utilizing waste paper to produce nanocellulose can reduce carbon emissions by up to 30% compared to nanocellulose production from other sources. However, challenges in applying nanocellulose on an industrial scale still exist, including production costs, storage stability, and compatibility with other construction materials.

Therefore, further research is needed to overcome these constraints and optimize the production process and application of nanocellulose in mortar. Collaboration between researchers, industry, and government is also important to encourage the widespread adoption of this technology in the construction sector [9]. In conclusion, nanocellulose synthesized from waste paper at low temperatures offers an innovative and sustainable solution to improve the microstructural properties of mortar. Its potential to reduce porosity, increase compressive strength, and reduce environmental impact makes it an attractive additive for the construction industry. With the continued development of technology and research in this field, nanocellulose is expected to become integral to global efforts to build a greener and more sustainable future. The main problem underlying this research is the lack of understanding of the effect of waste paper-based nanocellulose on the microstructure of mortar. In recent years, attention to environmentally friendly and sustainable materials has increased, especially in the construction industry. One material with great potential is nanocellulose, which can be obtained through a synthesis process from waste paper. Nanocellulose has unique properties, such as high mechanical strength, large surface area, and the ability to improve the properties of composite materials [10].

However, despite its potential, studies on the effects of nanocellulose on mortar, especially in terms of its microstructure, are still limited. Nanocellulose can be synthesized through various methods, including chemical and physical methods. The low-temperature synthesis process offers advantages in terms of energy efficiency and cost, making it a more sustainable option compared to other methods that require high temperatures. A study by [11] showed that nanocellulose synthesized at low temperatures can maintain its unique structure and characteristics without losing mechanical properties.

Thus, nanocellulose synthesized at low temperatures has excellent potential for use in construction applications. In the context of mortar, the microstructure of the material plays a vital role in determining the mechanical properties, durability, and water absorption capacity. The addition of nanocellulose is expected to improve the mortar microstructure by increasing interparticle bonds and reducing porosity. A previous study by [12] found that the addition of nanocellulose to mortar can increase microstructural density, reduce cracks, and improve resistance to water penetration. However, the study did not specifically review nanocellulose synthesized from waste paper at low temperatures, so there is still a gap in the literature that needs to be bridged. Waste paper as a raw material for nanocellulose provides dual benefits, namely waste reduction and the creation of value-added materials. According to [13], waste paper contains a relatively high amount of cellulose, making it very suitable for processing into nanocellulose.

This recycling process is also in line with the principle of the circular economy, which emphasizes the importance of maximizing resource use and minimizing waste. In this context, the synthesis of nanocellulose based on waste paper at low temperatures not only provides a sustainable material solution but also contributes to better waste management. This study aims to explore the effect of nanocellulose from waste paper on the microstructure of mortar. The main parameters analyzed include porosity, density, pore size distribution, and mechanical properties. The results of this study are expected to provide new insights into how to improve mortar performance by using nanocellulose as an additive. In terms of porosity, the addition of nanocellulose can contribute to reducing the number and size of pores in mortar. This is due to the ability of nanocellulose to act as a filler in the mortar matrix, thereby reducing the void space between particles. For example, a study by [14] showed that nanocellulose can fill small pores in concrete, which in turn increases resistance to water and chloride ion penetration.

The study also showed that nanocellulose can function as an additional binding agent, which strengthens the interparticle bond in the mortar. Pore size distribution is also an essential factor affecting the microstructural properties of mortar. The addition of nanocellulose can produce a more homogeneous pore size distribution, which contributes to improved mechanical properties and durability. According to the results of a study by [15], nanocellulose can regulate the pore size distribution by reducing the presence of large pores that are often the source of cracks. With a more uniform pore distribution, the mortar becomes more resistant to mechanical loads and environmental changes. In addition, the density of the mortar microstructure can also be increased by the addition of nanocellulose. Nanocellulose, which has a large surface area, can increase the bond between the particles that make up the mortar, resulting in a denser structure. This is in line with the findings of [16], who reported that mortar added with nanocellulose showed a 15% increase in density compared to conventional mortar. This increase in density also has an impact on reducing permeability and increasing resistance to damage due to freeze-thaw cycles.

However, the use of waste paper-based nanocellulose in mortar also has challenges. One of them is the compatibility between nanocellulose and other mortar ingredients. Several studies have shown that the addition of excessive amounts of nanocellulose can cause particle segregation and decrease mechanical properties [17]. Therefore, it is essential to determine the optimal proportion of nanocellulose to achieve the best performance. From an environmental perspective, the use of waste paper-based nanocellulose also has a significant positive impact. By recycling waste paper into nanocellulose, carbon emissions generated from the construction material production process can be reduced. In addition, the synthesis of nanocellulose at low temperatures consumes less energy than conventional methods, making it more environmentally friendly.

Research by [18] showed that the production of nanocellulose from waste paper resulted in a 30% lower carbon footprint compared to the production of nanocellulose from primary raw materials. Nanocellulose, as an environmentally friendly nanomaterial, has attracted significant attention in building materials research due to its excellent mechanical properties and ability to improve the microstructure of composite materials. One of the promising sources of nanocellulose is waste paper, which can be synthesized at low temperatures, offering an efficient and sustainable method for nanocellulose production. The synthesis process of nanocellulose from waste paper at low temperatures involves the use of a water-based solvent system such as NaOH/thiourea/urea. This method is effective in producing spherical nanocellulose with a cellulose II-type crystal structure.

According to research conducted by [19], nanocellulose produced through this method has an average particle size of around 50 nm and a crystallinity index of 48.85%, which is lower than that of bleached pulp (64.42%). This decrease in crystallinity indicates an increase in the amorphous area, which can increase the interaction between nanocellulose and the cement matrix in mortar [3]. The integration of nanocellulose into the mortar mixture can affect the microstructure of the material. The addition of nanocellulose has the potential to increase the distribution of more homogeneous cement particles, reduce porosity, and improve the bond between components in the mortar. This can produce materials with higher mechanical strength and better durability [20]. In addition, the hydrophilic nature of nanocellulose allows for better interaction with the cement matrix, which can improve the rheological properties of the mortar mixture and facilitate the hardening process [21].

Further analysis of the effect of nanocellulose on the mortar microstructure has shown that the use of nanocellulose improves the mechanical properties of the material. Studies using scanning electron microscopy (SEM) revealed that the uniform distribution of nanocellulose in the cement matrix contributed to the reduction of microcracks and increased interparticle cohesion. These results indicate that nanocellulose can act as a nanofiller that improves the overall mechanical properties of the mortar [22]. In addition, durability tests showed that the addition of nanocellulose increased the resistance of the mortar to environmental factors such as high humidity and exposure to aggressive chemicals. This effect is obtained due to the ability of nanocellulose to increase the density of the microstructure and minimize the penetration of water and harmful ions [23]. Therefore, nanocellulose synthesized from waste paper not only offers a solution for waste management but also contributes to the development of more sustainable and high-performance building materials.

This study highlights the novelty of utilizing low-temperature synthesized nanocellulose derived from waste paper as a sustainable additive for improving the microstructural properties of mortar. Unlike previous studies that primarily focused on general nanocellulose or high-temperature synthesis, this research emphasizes the effect of low-temperature processing on particle morphology, reactivity, and its direct influence on mortar porosity and density. The integration of this synthesis approach and its detailed microstructural evaluation provide new insights into developing high-performance, eco-friendly construction materials with reduced environmental impact.

2. Materials and Method

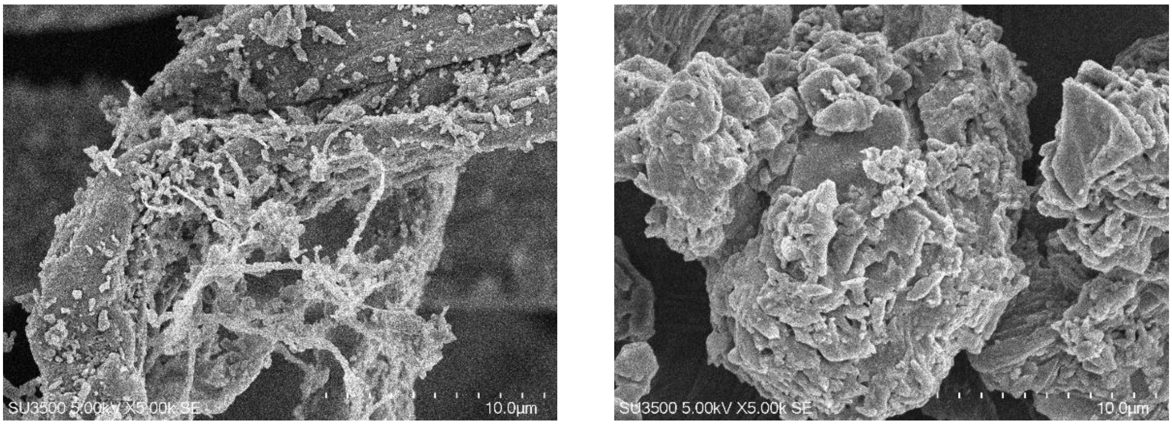
2.1. Materials

In this study, the mortar was prepared using Ordinary Portland Cement (OPC) type I material obtained from Cement Baturaja, with an average particle size between 1-100 µm and a specific gravity of 3.15, in accordance with the standards for high-quality cement materials. As a fine aggregate, the sand used came from Ogan Ilir, with an organic content of 2% and a mud content of 1.149%, which was chosen because of its physical and chemical properties that support the stability of the mortar mixture. The water used came from the Regional Drinking Water Company with a neutral pH of around ±7, ensuring its compatibility with other materials.

As an additional material, a superplasticizer was used to improve the workability of the mortar and reduce water requirements by 10%, resulting in a material with better mechanical properties. This study also involved nanocellulose synthesized from waste paper. The synthesis process is carried out using sodium hydroxide (NaOH) and sodium hypochlorite (NaClO) at low temperatures to maintain the authenticity of cellulose fibers. It produces nanocellulose particles with a size of around 100-500 nm and a more reactive surface.

These characteristics make nanocellulose potentially improve the mechanical properties and performance of mortar. As a comparison material, cellulose fibers from waste paper without special treatment and cellulose ash produced from burning waste paper were used. The purpose of this study was to evaluate the effect of nanocellulose (NC), cellulose ash (CA), and cellulose fiber (CF) on the quality and performance of mortar. Nanocellulose with a small particle size and a reactive surface is expected to increase the strength and durability of mortar.

In contrast, cellulose ash and untreated cellulose fiber provide a reference for comparing results with different levels of modification. The nanocellulose synthesis process using alkali materials ensures efficiency in processing waste paper into high-value additives, supporting environmental sustainability. This study aims to produce high-quality mortar by mixing various materials with different characteristics. The use of nanocellulose and its variations offers innovation in material technology and optimization of industrial waste utilization. This combination of materials is expected to contribute to the development of environmentally friendly, efficient, and optimally performing mortar for construction applications.



(a) (b)

Fig. 1. The morphology of the synthesized nanocellulose (a)Low temperature and (b)High temperature

2.2. Methods

The mortar mixing methodology in this study was designed to evaluate the effect of waste paper-based nanocellulose synthesized at low temperatures on the microstructural properties of the mortar. The mixing process was carried out systematically to ensure material homogeneity and accurate testing results. The stages of the mixing methodology, starting from the preparation of the materials used, include Ordinary Portland Cement (OPC) type I, fine sand from Ogan Ilir Regency, water from the Regional Drinking Water Company, superplasticizer, nanocellulose, cellulose fiber, and cellulose ash from waste paper. OPC cement with a specific gravity of 3.15 was chosen to ensure the quality and consistency of the mortar mixture.

The sand was sieved using a standard sieve to obtain a uniform particle size distribution, with mud and organic content that had been tested according to the ASTM C33 standard. Natural mineral water was used because it has a neutral pH (pH 7), which will not affect the chemical reactivity in the mixture. Superplasticizer was added to improve the workability of the mortar mixture while reducing water requirements by up to 10% compared to standard mixtures. Nanocellulose is synthesized from waste paper using sodium hydroxide (NaOH) and sodium hypochlorite (NaClO) solution at low temperatures. This process produces nanocellulose with a particle size of about 100-500 nm. As a comparison, cellulose fiber from waste paper without chemical treatment and cellulose ash produced from burning waste paper were used. These three materials, including nanocellulose, cellulose fiber, and cellulose ash, were prepared in sufficient quantities according to the planned mixture proportion.

Mortar mix design the mortar mix design follows the mix design standard that has been adjusted for this study. The ratio of cement, sand, and water is 1:2.75:0.5 based on the weight of cement. In this study, nanocellulose, cellulose fiber, and cellulose ash were added to the mixture in certain proportion variations, namely 0% and 2% of the weight of cement. This variation was carried out to evaluate the effect of the concentration of additional materials on the properties of the mortar, as seen in Table 1. Superplasticizer was added in a fixed amount of 1.0% of the weight of cement in each mixture variation to ensure optimal workability. The mortar mixing process is carried out through several systematic stages to ensure an even distribution of materials and optimal mixture quality. The first stage begins with a dry mixing of cement and sand. Both are measured according to the designed proportions and then mixed in dry conditions using a mechanical mixer for two minutes. This step aims to obtain initial homogeneity between cement and sand before adding water.

Next, the water that has been measured and mixed with the superplasticizer is stirred manually until completely dissolved, forming a homogeneous solution. This solution is then added slowly to the dry mixture while stirring using a mixer at low speed until the mixture begins to form a paste. After that, additional materials such as nanocellulose, cellulose fibers, or cellulose ash are added to the mixture according to the designed proportion variations. These additional materials have previously been stirred manually to break down agglomeration that may occur during storage. After being added, the entire mixture is stirred again at medium speed for three minutes until the mixture becomes homogeneous. After the mixing process, the flow workability of the mortar mixture is checked using the flow cone test according to the ASTM C1437 standard. If the mixture has a workability outside the desired range, the amount of water or superplasticizer will be adjusted until the workability meets the criteria. After workability is achieved, the mixture is stirred again at high speed for two minutes to ensure that all materials are thoroughly mixed and no segregation occurs.

At the molding stage, the homogenized mortar mixture was poured into a cube-shaped mould measuring 50 x 50 x 50 mm according to the ASTM C109 standard. Before being filled, the mould was coated with lubricant so that the sample could be easily removed. Each mould was filled in three layers, and each layer was compacted using a pounding rod to remove trapped air. After the molding process was complete, the samples were left for 24 hours at room temperature (25±2°C) for initial curing. Furthermore, the samples were removed from the mould and immersed in a curing tank filled with clean water at the same temperature until they reached the test age of 28 days to ensure that the hydration process was complete. Microstructural analysis is an important stage in mortar research, especially to understand the physical and chemical characteristics that contribute to the performance and sustainability of the material. Through microstructural analysis, it can be seen how the distribution, shape, and interaction between particles in the mortar are, including the identification of phases formed due to chemical reactions during the hardening process. In addition, this analysis also allows for the evaluation of the effectiveness of the additional materials used, such as nanocellulose, cellulose fibers, or cellulose ash, in improving the internal structure of the mortar.

The analysis was carried out comprehensively by integrating several complementary advanced characterization techniques. The Energy Dispersive X-ray (EDX) technique was used to identify and map the composition of chemical elements in the mortar. EDX provides quantitative information on elements such as calcium, silicon, aluminum, and others relevant to the formation of cement hydration products. Furthermore, the Scanning Electron Microscope (SEM) was applied to obtain a detailed visual image of the surface structure and morphology of mortar particles on a micrometer to nanometer scale. SEM provides high-resolution images that allow observation of the shape, size, pore distribution, and homogeneity level of the mortar mixture, as well as identification of cracks or microcavities that can affect the strength of the material. In addition, the X-ray diffraction (XRD) technique was used to analyze the mineral phases and crystallography formed in the mortar during the hydration process. XRD can identify the types of crystalline compounds formed, such as calcium silicate hydrate (C-S-H), calcium hydroxide, and unreacted raw material residues, thus allowing the assessment of chemical reactions and the efficiency of the hydration process.

Fourier Transform Infrared Spectroscopy (FTIR) is used to determine the chemical functional groups contained in mortar. FTIR provides spectrum data that indicates the presence of certain chemical bonds, allowing it to detect changes in chemical composition due to the addition of additives or during the hydration process.

By combining the four techniques, the research was able to produce a more holistic understanding of the internal structure and chemical composition of mortar. This integrated microstructural analysis is the basis for developing more efficient, high-performance, and environmentally friendly mortar materials according to the demands of today's sustainable development. Each technique has an important role in providing information about the chemical composition, crystallinity phase, microstructure, and interactions between materials in mortar. FTIR is a technique used to identify functional groups in materials and to study the chemical changes that occur as a result of material changes in mortar. FTIR works by reducing infrared light at various wavelengths, which causes molecules in the material to vibrate at specific frequencies according to their chemical properties. XRD is a technique used to identify crystalline phases in solid materials and is very important in analyzing the crystal structure of mortar. XRD works by sending X-rays into a sample. When the X-rays hit the sample, some of the X-rays will be reflected by the crystal layer at certain angles. The resulting X-ray reflection pattern can be analyzed to determine the crystal structure, crystal grain size, and crystal orientation in the material. SEM is a microscopic technique used to study the microstructure of materials at a very detailed level. SEM produces images of a sample's surface by utilizing electrons reflected or transmitted from the sample.

In addition to providing a visual representation of the surface structure, SEM also allows for local analysis of the material composition. In the context of mortar analysis, SEM is used to observe the distribution of nanocellulose, cellulose fibers, and cellulose ash in the mortar matrix. EDX is an analytical technique used to analyze the chemical elements in a sample by detecting the X-rays emitted by atoms in the sample after they are bombarded with electrons. Each chemical element has a characteristic energy for the X-rays emitted, thus allowing the identification of the elements contained in the sample.

2.3. Characterization of Mortar Nanocellulose

Characterization of nanocellulose is an important step in understanding the physical and chemical properties of this material as a whole. One of the primary methods used is Fourier Transform Infrared Spectroscopy (FTIR), which functions to identify functional groups in nanocellulose. FTIR is also able to detect chemical changes that occur due to specific modifications or treatment processes, thus providing an overview of the chemical composition and molecular interactions in nanocellulose. In addition, analysis using X-ray Diffraction (XRD) is essential to reveal the crystal structure of nanocellulose.

Fig. 2. Test specimen manufacturing procedure

XRD can identify the crystalline phase and provide data on the level of crystallinity and crystal grain size, which play an important role in determining the strength and mechanical stability of the material. Observation of the morphology of nanocellulose was carried out using a Scanning Electron Microscope (SEM). Through SEM, the surface and microstructure of nanocellulose can be visualized in detail, showing the distribution of fibers and the interaction of nanocellulose with the matrix or other materials at the microscopic level. An Energy Dispersive X-ray (EDX) was used to analyze the composition of chemical elements. EDX measures the elements contained in nanocellulose by detecting X-rays emitted when the sample is bombarded with electrons, providing quantitative data on the elemental composition.

In addition, the particle size distribution of nanocellulose was also analyzed because it dramatically affects the material's performance and application, especially in the construction field, where particle size plays a role in mechanical properties and compatibility with other materials. Combining these characterization methods provides a comprehensive picture of the quality and potential of nanocellulose for various industrial applications. In Fig. 2, the flow and process of making research samples are shown.

Table 1. Mixing proportions for cementitious mortar (kg/m^3)

Mixes	OPC	Fine Aggregate	Water	Cellulose
N	666,67	1833,33	299,99	0
NC	653,33	1833,33	299,99	4
CA	653,33	1833,33	299,99	4
CF	653,33	1833,33	299,99	4

3. Result and Discussion

3.1. Flow Testing

Mortar flow testing is one of the essential methods in determining the quality and characteristics of mortar for construction applications. Mortar is a mixture of materials that bonds bricks, stones, or other building materials. Mortar dryness or workability is one of the crucial aspects because it affects the ease of application, strength, and resistance of materials to deformation or cracking after application. Fig. 3 shows the flow test results.

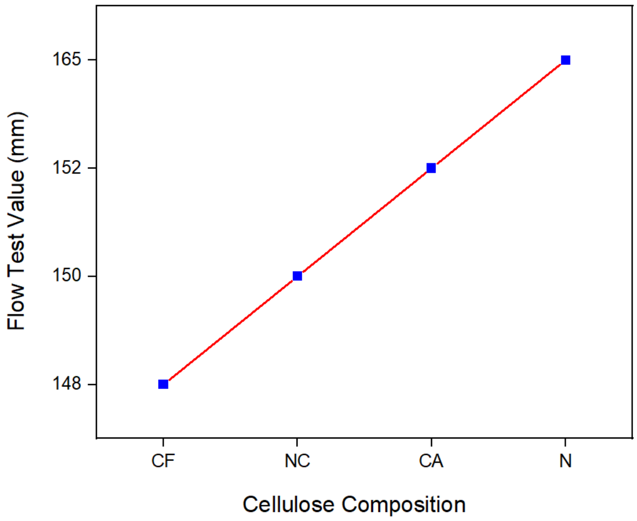


Fig. 3. Effect of cellulose composition on flow test values

Friction testing is usually performed using standard methods, such as the flow table test (ASTM C1437). In this method, fresh mortar is placed on a cone-shaped flow table, then stomped several times to measure the change in the diameter of the mortar. This test provides an overview of the viscosity and fluidity of the mortar mixture. Based on Fig. 3, the flow test value for the CF composition (2% cellulose fiber) is 148 mm, for the NC composition (2% nanocellulose) is 152 mm, the CA composition (2% cellulose acetate) yields 158 mm, and the N composition (0% fiber) reaches the highest value of 165 mm. These results clearly indicate that the flow diameter decreases as the fiber content increases, showing that the incorporation of 2% cellulose-based materials reduces the fluidity of the mortar.

This reduction in flowability can be attributed to the physical interaction between the cellulose fibers and the cement matrix. The fibers tend to absorb part of the mixing water due to their hydrophilic nature, which decreases the amount of free water available for lubrication among particles. In addition, the entanglement of fibers increases internal friction within the mix, leading to higher viscosity and lower mobility of the mortar. Among the fiber-reinforced mixes, the CA sample shows a slightly higher flow (158 mm) compared to NC and CF, possibly due to the smoother surface and lower water absorption of cellulose acetate fibers, which allow better dispersion in the mix. Conversely, CF and NC exhibit lower flow values (148–152 mm) because their rougher and more reactive surfaces promote stronger interaction with the cement paste, increasing the cohesiveness of the mixture.

Based on research published by [24] in Construction and Building Materials, the elasticity of mortar is influenced by various factors such as the proportion of constituent materials (cement, sand, and water), the type of additives used, and the mixing method. The main factors determining the degree of adhesion are the correct proportion of materials and the consistency of the mortar mixture. For example, the water-to-cement ratio (w/c ratio) plays a vital role in increasing adhesion, but too high a ratio can reduce the strength of the mortar after hardening.

The addition of additives, such as superplasticizers, also has a significant effect on the elasticity of the mortar. According to [25], this additive can increase elasticity without adding excessive water. Superplasticizers work by evenly dispersing cement particles, thereby reducing the mixture's viscosity and increasing its fluidity. These additives are particularly relevant in applications where surface quality and mortar adhesion are priorities. Furthermore, the elasticity of mortar is also greatly influenced by the physical properties of its constituent materials, especially sand. The size of the sand grains, the distribution of gradations, and the shape of the particles play an important role. According to [26] in the Journal of Materials in Civil Engineering, sand with good gradation provides optimal contact between particles, resulting in higher friction. On the other hand, sand with too large granules or uneven distribution can cause the mixture to become stiff and difficult to apply.

The ambient temperature also affects the adhesion of the mortar. According to [27] in the Journal of Materials in Civil Engineering, mortars mixed at high temperatures tend to lose their adhesion faster due to the acceleration of cement hydration. This requires the adjustment of composition or the use of certain additives to maintain the elasticity of the mortar, especially in hot climate applications. In addition to flow tables, other slip test methods, such as slump cones, can be used, especially for high-consistency mixtures. According to [28], the slump cone test is more suitable for dry mortar or low moisture content. It provides information about the ability of the mortar to maintain its shape on both vertical and horizontal surfaces.

Good dryness in the early stages of application is essential to ensure even distribution of materials, reducing the risk of air cavities or micro-cracks that can degrade the strength and durability of the structure. According to [29], fatigue testing is essential to ensure ease of application and guarantee the quality and longevity of the construction finish. In modern construction practices, automated testing tools and sensor-based technologies have begun to be applied to improve the accuracy and efficiency of mortar friction testing. According to [30], it can measure the viscosity and viscosity of mortar in real-time, producing more detailed and comprehensive data than manual methods. In addition to the technical aspect, elasticity testing also plays a vital role in developing environmentally friendly materials. According to [31], cement substitutes such as fly ash and slag can increase elasticity while reducing carbon footprint. These materials not only provide good fluidity but also support the sustainability of the construction industry. Overall, mortar fatigue testing is essential in ensuring the quality, performance, and sustainability of construction materials. Factors such as the ratio of materials, type of additives, physical characteristics of the sand, and environmental conditions must be considered thoroughly. As technology and material innovation develop, mortar elasticity testing methods will continue to evolve, opening up opportunities to create more efficient, high-performance, and environmentally friendly materials. A multidisciplinary approach is essential in understanding and optimizing mortar elasticity for the efficiency and sustainability of the construction industry of the future.

3.2. Compressive Strength

Mortar compressive strength testing is one of the important methods in determining the quality and characteristics of mortar for construction applications. Mortar is a mixture of materials used to bond bricks, stones, or other building materials. Compressive strength is a crucial aspect because it has a direct effect on the strength of the structure, resistance to loads, and longevity of construction. The results of the compressive strength test can be seen in Fig. 4.

The results of compressive strength tests on various cellulose compositions can be seen from Fig. 4, which shows the relationship between the material's composition and the mortar's compressive strength. In the graph, the cellulose composition labeled N shows the highest compressive strength value, 28.72 MPa, followed by the compositions NC, 30.16 MPa, CA, 26.54 MPa, and CF, 23.51 MPa. This shows that the higher the cellulose content in the mortar, the greater the ability of the mortar to withstand compressive loads. This higher composition suggests that the quality of the binding materials used in the mortar mixture plays a vital role in improving pressure resistance, which has implications for the durability of the structure in the long term.

The relatively high compressive strength of 28 MPa in the normal mortar, even without coarse aggregate, can be explained by several factors. First, the fine aggregate (sand) used has good gradation and surface characteristics, which enhance the packing density and reduce voids in the matrix. This leads to a denser microstructure and higher compressive capacity. Second, the water-to-cement (w/c) ratio was maintained at an optimal level, allowing sufficient hydration of cement without producing excessive pores. Third, the curing process was carried out under controlled conditions, ensuring complete hydration and uniform hardening throughout the specimen. These combined factors result in a compact and strong cement paste–sand matrix, allowing the mortar to reach a compressive strength comparable to that of normal concrete mixtures with low aggregate content. Similar findings have been reported by [32] and [33], which noted that optimized mix proportion and good material gradation could produce mortar compressive strengths in the range of 25–30 MPa even without coarse aggregates.

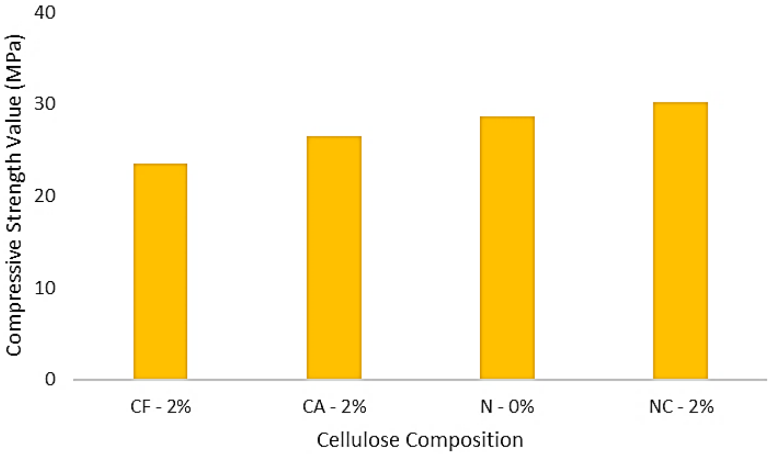


Fig. 4. Effect of cellulose composition on compressive strength of mortar

Based on research by [32] in Construction and Building Materials, the compressive strength of mortar is greatly influenced by the composition of constituent materials such as cement, sand, water, the type of additives, and the mixing method. Proper material comparison and mixture consistency are essential in determining how much compressive strength can be achieved. One example is the ratio of water to cement (w/c ratio), where adding water makes mixing easier. Still, excess water reduces the compressive strength of the mortar after the hardening process.

The most common compressive strength testing method is a cube or cylindrical compressive test (ASTM C109/C109M), where a press will test a pre-molded mortar sample until it breaks. This process aims to determine the maximum load the mortar can withstand before it is damaged. The results of the compressive strength test of this mortar may vary depending on the addition of additives. For example, mortar without additives generally has a lower compressive strength than mortar with the addition of certain materials. Research by [33] suggests that using superplasticizers as additives can increase compressive strength without adding excess water, because this material distributes cement particles more evenly, resulting in a denser and stronger mixture. In addition to the composition and additives, the physical characteristics of constituent materials, such as sand, also affect the compressive strength of the mortar. Sand with good gradation and appropriate particle shape will produce optimal contact between particles, resulting in a denser and stronger mixture. Conversely, sand that is not uniform or too large can cause air cavities in the mix, decreasing the compressive strength. This has been proven by [34], which revealed that finer and more uniform sand produces stronger mortars.

Environmental factors, particularly temperature during mixing and hardening, also greatly affect the compressive strength of the mortar. Research by [35] shows that high temperatures can speed up the cement hydration process so that the mortar hardens quickly, but can reduce long-term compressive strength if not balanced with mixture adjustments or special additives. Therefore, temperature regulation is essential in the mortar production process, especially in countries with hot climates. In addition, mortar compressive strength testing is essential to determine the material's resistance to loads and structural strength. A good result in the pressure test indicates that the material is highly resistant to damage or cracking. Thus, the compressive strength testing of mortar is important not only for the application stage but also to ensure the quality and durability of the construction finish.

With the development of technology, sensor-based mortar compressive strength testing tools and automated systems are now starting to improve the accuracy and efficiency of testing. These modern devices allow for real-time measurement of compressive strength and provide more detailed data than manual testing, as developed by [36] in Advanced Industrial and Engineering Polymer Research. This technology brings significant changes in the testing of construction materials, improves testing quality, and opens up opportunities for developing more environmentally friendly and efficient materials.

3.3. Fourier Transform Infrared Spectroscopy (FTIR)

In recent years, FTIR (Fourier Transform Infrared Spectroscopy) has gained popularity due to its increased sensitivity and applications in various fields, including nanotechnology and environmental analysis. Recent studies have shown that they can analyze materials at the molecular level with higher accuracy, even for small sample sizes [37]. FTIR is also increasingly used in environmental quality assessments, such as air and air pollutant detection [38]. FTIR analysis for biomaterial and pharmaceutical sensing also has significant implications for clinical and pharmacological research [39]. The FTIR test result spectrum of cellulose mortar can be seen in Fig. 5.

Fig. 5 This spectrum shows the sample's FTIR test result, which shows a specific energy absorption frequency zone connected to the vibration of molecular chemical bonds. In recent years, Fourier Transform Infrared Spectroscopy (FTIR) has increased in popularity as a highly sensitive and applicable analytical method in various fields, including nanotechnology and environmental analysis. The development of FTIR technology allows material analysis at the molecular level with higher accuracy, even for small samples [37]. In the environmental field, FTIR is now widely used in environmental quality assessment, such as the detection of air pollutants, both in the gas and particulate phases [38]. FTIR applications are also increasingly widespread in the fields of biomaterial and pharmaceutical sensing, which have significant implications for clinical and pharmacological research [40].

The FTIR spectrum results of standard samples (N) generally show a frequency zone that reflects the vibration of certain molecular chemical bonds. For example, the peak at around 3300 cm^{-1} is usually associated with O-H bond vibrations, while the peak at around 1700 cm^{-1} indicates C=O vibrations. Through in-depth analysis, these peaks can be used to identify functional groups and molecular structures in the material being analyzed. The distribution and shape of these absorption peaks help determine the chemical composition of the sample. Recent innovations in FTIR technology allow for better analysis of smaller sample sizes and materials with high complexity [40,41].

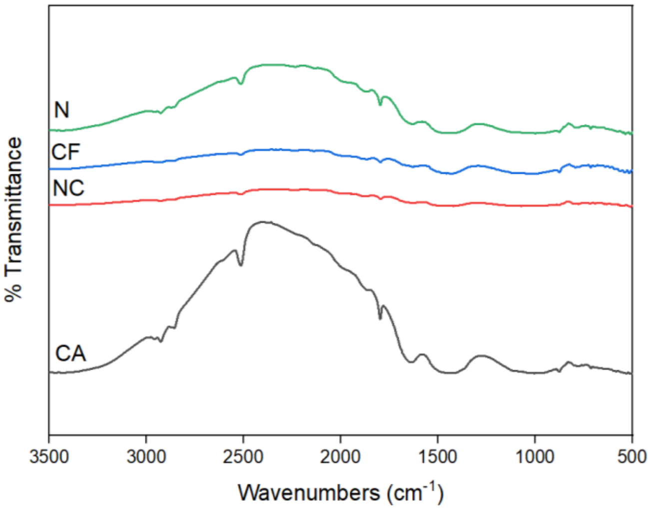


Fig. 5. FTIR test result spectrum of mortar

In the FTIR spectrum of cellulose fiber (CF), the characteristic peaks detected at around 3300 cm^{-1} and 1700 cm^{-1} are related to the O-H and C=O groups, respectively. The presence of these bands is important to assess the purity of organic compounds used in the material. The use of FTIR has skyrocketed, ranging from pharmaceutical testing and food quality analysis to environmental monitoring. FTIR is considered very effective in identifying environmental contaminants, especially in the air [42], and has improved clinical diagnostic capabilities, especially in biomaterial sensing [43]. Nanocellulose (NC) displays several unique peaks in the FTIR spectrum, indicating the presence of both organic and inorganic functional groups. O-H or N-H stretching generally causes a dominant peak around 3300 cm^{-1}, which represents amines, phenols, or alcohols. Carbonyl (C=O) stretching also typically appears around 1700 cm^{-1}, indicating the presence of carboxylic acids, aldehydes, or ketones in the material structure [44].

The FTIR spectrum of cellulose ash (CA) also shows important peaks, especially at 3300 cm^{-1} (O-H stretching), as well as a peak around 1700 cm^{-1} (C=O stretching), which is usually found in alcohols, phenols, or carboxylic acids. The presence of these peaks confirms the chemical composition of cellulose ash, which is rich in these functional groups [45]. In general, the FTIR spectrum displayed shows typical absorption peaks related to specific chemical vibrations. The O-H or N-H stretching, which usually originates from hydroxyl or amine groups, is clearly visible at peaks around 3300 cm^{-1}. This indicates the presence of amines, phenols, or alcohols in the sample. Meanwhile, the peak around 1700 cm^{-1} is a reliable indicator of the C=O stretching vibration of carbonyl groups such as those found in carboxylic acids, ketones, or aldehydes. In addition, the C-H stretching, both in aliphatic and aromatic hydrocarbons, is usually seen around 2900 cm^{-1}, and the CH_2 bending vibration gives a peak around 1450 cm^{-1}, indicating the presence of alkyl groups [46].

The FTIR spectrum also often shows other significant peaks and valleys, for example, at 3480 cm^{-1}, 2925 cm^{-1}, 1736 cm^{-1}, 1639 cm^{-1}, and 1422 cm^{-1}, all of which are related to various chemical bond vibrations in the material structure. The deepest valley around 3500–3000 cm^{-1} indicates low transmittance or high absorption in that region. In general, transmittance will increase (or absorption decreases) as the wave number decreases, although there are still fluctuations caused by differences in molecular structure [47]. The addition of nanocellulose to mortar is known to affect the intensity of these peaks, especially increasing the intensity of the O-H peak, indicating a stronger interaction between nanocellulose and the cement matrix, which can increase the strength of the mortar [48].

The FTIR spectrum can be visualized as a graph with the x-axis as the wave number (cm^{-1}) from 4000 to 500 and the y-axis as the per cent transmittance. The absorption pattern formed reflects the chemical content and molecular structure of the material. The red curve commonly used in the illustration shows the specific pattern of each sample. The prominent peaks that are often observed are at around 3464 cm^{-1} (O-H or N-H), 2912 cm^{-1} (C-H), and the peak below 1500 cm^{-1}, which is known as the fingerprint region, where each compound tends to have a different characteristic pattern [49]. The sharp peak at 1736 cm^{-1} is usually associated with the carbonyl group (C=O), and below 1500 cm^{-1}, many small peaks are significant for the specific identification of the molecular structure [50]. A broad and strong peak around 2912 cm^{-1} is usually a sign of C-H bonds, for example, in methyl or methylene groups [51]. In this region, changes in peak intensity or position can be an indication of interactions between additives such as nanocellulose and other components in the mortar.

In the practice of FTIR analysis on nanocellulose mortar, samples are usually prepared in the form of thin films or pellets through a drying and grinding process. The samples are then analyzed with an FTIR spectrometer that exposes the infrared light spectrum and records the absorption pattern at various wavelengths. The resulting spectrum is then analyzed to identify characteristic peaks corresponding to chemical bonds, such as O-H stretching in the range of 3200–3600 cm^{-1}, C-H stretching in the range of 2800–3000 cm^{-1}, C=O stretching around 1700 cm^{-1}, and C-O stretching and O-H bending at 1000–1200 cm^{-1} [51],[52].

Interpretation of the FTIR spectrum is based on the recognition of specific peaks corresponding to functional groups found in nanocellulose and the mortar matrix. In addition, changes in the position or intensity of peaks, both in pure nanocellulose and in mortar, can be an indication of interactions between nanocellulose components and cement, such as the formation of new hydrogen bonds or the occurrence of chemical reactions [53]. This spectrum also helps to determine whether the nanocellulose additive has been well dispersed in the mortar matrix or has actually clumped.

In pure nanocellulose, the O-H stretching peak usually appears in the range of 3200–3600 cm^{-1}, the C-H stretching at 2800–3000 cm^{-1}, and the C-O stretching at 1000–1200 cm^{-1}. In mortar without nanocellulose, the FTIR spectrum is generally dominated by peaks from cement hydration products, such as calcium silicate hydrate (C-S-H) [54]. Meanwhile, in a mortar containing nanocellulose, the peaks that appear are a combination of characteristics of both components, and any changes or shifts in these peaks can indicate interactions or the formation of new chemical structures between nanocellulose and cement. The practical implications of using FTIR in mortar research are enormous. FTIR analysis facilitates the optimization of nanocellulose content to improve the mechanical properties and durability of mortar, as well as ensure the quality and consistency of the performance of the modified material. Through the identification of molecular interactions and chemical structure changes, FTIR drives innovation in the development of advanced construction materials that have superior properties, are environmentally friendly, and are more adaptive to modern engineering needs.

Some important peaks that are often used as references are around 3400 cm^{-1}, which indicates the -OH group; the peak at 2900 cm^{-1}, which is related to the C-H bond of nanocellulose [55]; the peak at 1640 cm^{-1} which indicates bound water in the mortar [56], and the peaks at 1420 cm^{-1} and 875 cm^{-1} which are related to carbonate from cement hydration products [57]. The peak at 1100 cm^{-1} indicates the Si-O bond of Calcium Silicate Hydrate (C-S-H) [58]. The addition of nanocellulose can strengthen the intensity of the -OH and C-H peaks, thus confirming the interaction between nanocellulose and the cement matrix, which ultimately has the potential to improve the mechanical properties of the mortar [59]. Thus, FTIR becomes an essential analytical tool in the characterization and development of nanocellulose-modified mortars. This method provides deep insights into the chemical composition and interactions, which are crucial for optimizing the performance of mortars in advanced construction applications.

3.4. X-Ray Diffraction (XRD)

A powerful and frequently used analytical method to determine the crystal structure of a material is X-ray diffraction (XRD). The basic principle of XRD is to direct X-rays into a sample, where they interact with the crystal lattice of the material. X-rays that hit the crystal layer will be reflected, and the angle and intensity of the reflected light will provide important information about the internal structure of the crystal. The resulting diffraction pattern can be used to determine the distance between crystal layers, atomic arrangement, and other structural characteristics of the material.

XRD is essential in the material characterization process because it can provide detailed information about the crystal phase, grain size, and orientation of solid materials such as metals, ceramics, and polymers. This method has a vital role in various fields, such as materials science, pharmacological research, and geological studies. Knowledge of crystal characteristics is essential for creating new materials, evaluating existing materials, and improving material performance in real-world applications.

The spectrum of the XRD test results from cellulose mortar can be seen in Fig. 3. Each pattern has unique characteristics that reflect the composition and microstructural changes due to the addition of cellulose material in various forms [60]. In the XRD pattern of normal mortar (N), high-intensity peaks are clearly detected at several 2θ angle positions, which are commonly found in hydrated Portland cement products. These peaks indicate the presence of major crystalline phases such as alite (C_3S), belite (C_2S), and portlandite ($Ca(OH)_2$ or CH).

The presence of sharp peaks with prominent intensity reflects the high degree of crystallinity in these major hydrated phases. The significant peak intensities at around $2\theta \approx 29°$, 32°, and 34° can generally be attributed to CH and several calcium silicates phases. These peaks are important indicators that the hydration process in normal mortar is optimal and produces a proportion of hydrated products that meet the standards of conventional mortar.

Fig. 6 X-ray Diffraction (XRD) analysis is one of the main techniques in characterizing the crystal structure and mineral phase changes in cement-based materials, including mortars modified with nanocellulose. Through the XRD diffraction pattern, various main crystalline and amorphous phases in the material can be identified explicitly while observing the effect of adding additives such as nanocellulose on the evolution of the internal structure of the mortar [5],[61]. XRD diffraction patterns of several mortar variations, namely normal mortar (N), mortar with cellulose fiber (CF), mortar with nanocellulose (NC), and cellulose ash mortar (CA), in the 2θ angle range between 10° and 60°.

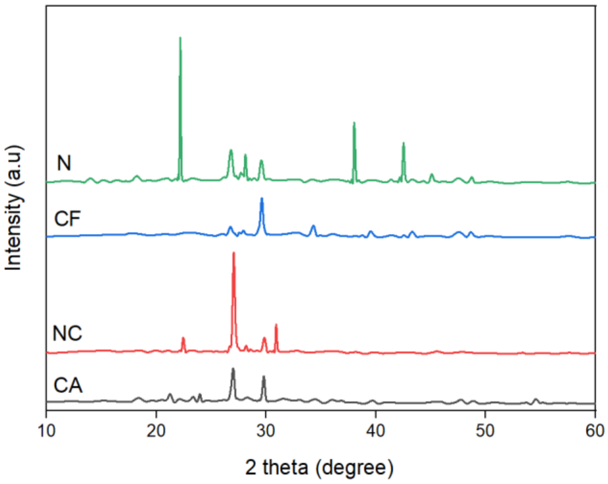


Fig. 6. XRD test result spectrum of mortar

The addition of cellulose (CF) fibers in the mortar reveals specific changes to the XRD pattern. Although the prominent peaks of the cement hydration phases are still visible, there is a slight decrease in intensity at some peaks, indicating the beginning of interaction between the organic components of the cellulose fibers and the cement matrix. These interactions tend to result in a higher amorphous phase distribution or lower levels of local crystallinity around the fibers, although overall, the main structure of the mortar remains dominated by the cement crystal phase. However, there were no significant new peaks in the CF sample, suggesting that the cellulose fibers in the concentrations used still act as fillers without significantly forming new crystalline phases.

More interesting results were seen in mortars modified with nanocellulose (NC). In the XRD pattern of NC samples, significant changes were found in the amorphous structure as well as the phase of the main crystal. One of the important findings is the appearance of sharper diffraction peaks at $2\theta \approx 22.6°$, which is characteristic of cellulose crystals. The peak intensity at this angle was much higher in the NC samples than in the N, CF, and CA. This is in line with the report of [62], where the peak of nanocellulose characteristics at $2\theta \approx 22.5°$ strengthens as the concentration of nanocellulose increases. This increase signifies an increase in the level of crystallinity in the mortar system due to the good dispersion of nanocellulose, as well as the role of nanocellulose as a new phase formation with cement hydration products. Furthermore, in NC mortars, an increase in peak intensity was also observed in the position attributed to hydration products such as Calcium Silicate Hydrate (C-S-H) and ettringite, which suggests that nanocellulose with low-temperature synthesis can promote the formation of dense secondary hydration products. The increase in peak intensity in this hydration product indicates a more optimal cement hydration process. Nanocellulose acts as a nucleation site that accelerates the formation and growth of C-S-H gel, thereby filling the micro-pores within the cement matrix and increasing the overall density of the mortar structure [63].

Another phenomenon identified from the XRD data is a decrease in calcium hydroxide (CH) crystallinity, as seen from the weakening of the CH peak intensity, especially at about $2\theta \approx 34°$. This suggests that nanocellulose induces pozzolanic reactions, where CH reacts with amorphous silica released by nanocellulose, producing more C-S-H. A decrease in CH also means less free $Ca(OH)_2$, which can cause adverse chemical reactions in aggressive environments, thereby increasing the resistance of the mortar to external chemical attacks [64]. Thus, the decrease in CH crystallinity is an important indicator of the synergistic reaction between nanocellulose and cement, which strengthens the performance of mortar. In addition to shifts or changes in peak intensity, XRD data on NC mortars also show a certain peak widening occurring. This peak dilation is generally associated with a decrease in crystalline grain size or a more even distribution of the semi-crystalline phase, resulting from good dispersion of nanocellulose in the cement matrix. The study of [61] showed that nanocellulose can interact with cement components to form denser tissues, reduce porosity, and improve the mechanical properties of the mortar, as reflected in a wider but intense diffraction pattern at some angles. Furthermore, the latest study also reports new peaks in the XRD pattern of NC mortars that are not present in the N, CF, or CA samples. These new peaks indicate the formation of new crystalline phases derived from the unique interaction between nanocellulose and the cement matrix. The emergence of these new phases is positively correlated with increased compressive strength, flexural strength, and resistance to microcracking [65].

This performance-enhancing effect is highly dependent on the concentration of nanocellulose used as well as how well nanocellulose is dispersed in the mixture [23]. When compared to cellulose ash (CA) mortar, the XRD pattern in CA shows primary peaks similar to normal mortar but with lower intensity and wider peak distribution. This indicates that cellulose ash tends to increase the amorphous fraction in the mortar while retaining some of the key characteristic peaks of the hydration product. There are no prominent cellulose crystal peaks, indicating that the organic phase has been heavily decomposed during the ash combustion process. In addition to being a quality control tool, XRD in this study also provides in-depth insights into how nanocellulose affects the microstructure properties and mechanical performance of mortars. The XRD pattern shows an increase in peak intensity at $2\theta \approx 22.5°$ and a decrease in CH peaks, confirming that nanocellulose acts not only as a filler but also as an active booster that accelerates hydration reactions, improves structural density, and forms a new phase that is more stable and robust [66]. These data reinforce the conclusion from various literature that the interaction between nanocellulose and cement can be maximized to produce mortar materials with superior mechanical properties and better environmental resistance.

The application of XRD in the production quality control of nanocellulose-based construction materials is also critical, as the consistent XRD diffraction pattern indicates that the production process has produced a material with a stable and optimal crystal structure [67]. Any anomalies in the diffraction pattern, whether in the form of unexpected new peaks or the loss of the central peak, can be immediately detected and used as a basis for further formulation improvements or production processes.

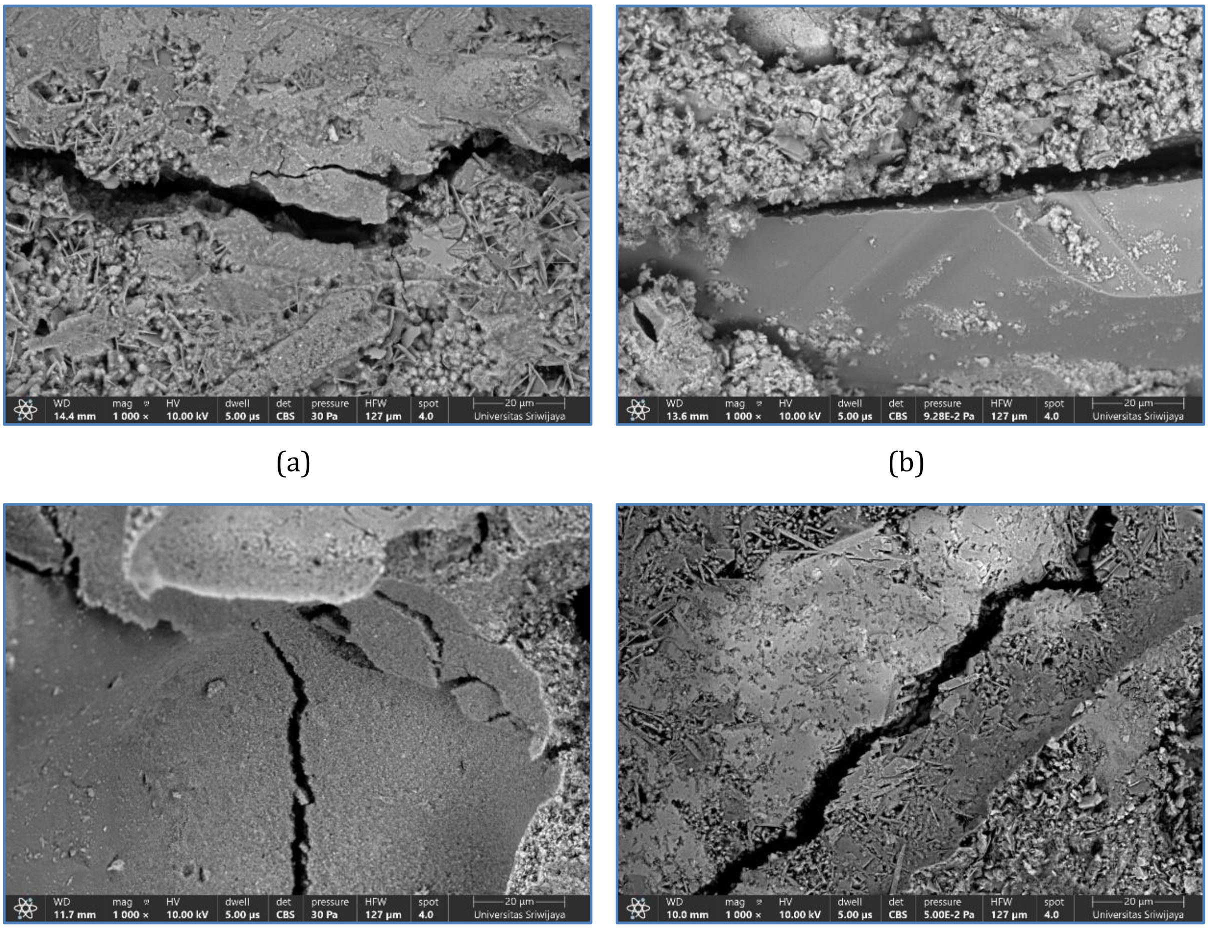
In the development of science, XRD analysis on nanocellulose mortar is an important foothold for environmentally friendly building material innovation. The XRD results provide empirical evidence that nanocellulose can interact synergistically with cement, accelerating the formation of secondary hydration products, lowering harmful phases such as CH, and increasing the proportion of beneficial crystalline structures. Thus, the resulting material not only has better technical performance but also contributes to the reduction in the use of pure Portland cement, thus supporting the principles of sustainability in the construction sector. The above XRD shows how variations in material composition, especially the addition of nanocellulose, significantly alter the crystal structure, the amorphous phase distribution, and the formation of hydration products in the mortar. XRD patterns not only reflect material properties at the microscopic level but also closely correlate with the results of mechanical properties testing, such as compressive strength, crack resistance, and environmental durability. Therefore, XRD analysis is a critical method in the research and development of innovative mortars, both for production quality control and for further scientific exploration of the interaction mechanisms of nanocellulose and cement at the atomic to microstructural levels. This data is also the basis for the development of more potent, more durable, and environmentally friendly nanocellulose-based building material formulations in the future.

3.5. Scanning Electron Microscope (SEM)

In recent years, SEM (Scanning Electron Microscopy) has gained popularity thanks to its extremely high-resolution capabilities and wide application in a wide range of fields, including materials science and biotechnology. SEM is also increasingly used in morphological analysis and material characterization, for example, to detect microstructural damage, observe particles, and evaluate pore and crack distribution in detail [68].

Fig. 7(a) Testing using Scanning Electron Microscopy (SEM) on normal (N) mortar showed the presence of separator cracks with no detected fibers. SEM allows high-resolution observation of mortar surfaces, thus providing detailed information about grain distribution, porosity, and the presence of microcracks in mortar samples [69]. This information is beneficial for evaluating the quality and resistance of mortars to environmental influences, such as humidity and temperature changes [70]. Previous research has shown that SEM can be used to examine the interactions between mortar constituent materials, such as cement, aggregate, and water, as well as to identify the effect of additive additions on material morphology [71]. In addition, SEM provides insights into pore distribution at the microscopic level that correlates with compressive strength and mortar resistance to freezing-thaw cycles [72,73]. Thus, the results of SEM testing can provide a deeper understanding of how the composition of mortar materials affects their structural performance under real-world conditions [74].

Fig. 7(b) SEM testing on mortars with the addition of cellulose fiber (CF), which is derived from waste paper and used as a cement composite material, showed an increase in the mechanical strength and durability of the material. SEM is used to analyze the distribution of fibers in a mortar matrix, as well as the interactions between cellulose fibers and the main components of mortar, such as cement, sand, and water [75]. With high resolution, SEM can reveal surface morphology in detail, including fiber thickness and orientation, as well as the presence of cracks or defects in microstructures that can affect material performance. Research by [76] shows that the addition of cellulose fibers can increase tensile strength and resistance to crack propagation, which is reflected in a more homogeneous surface structure of the mortar and better cohesion between components. SEM is also used to evaluate the distribution of fibers in cement matrices, so researchers can identify potential problems in the mixing and compaction process that could affect the final result of the mortar [77]. In addition, SEM was used to study the effect of variations in the proportion of cellulose fibers on the micro properties of mortars, including resistance to freezing-thaw cycles and corrosion [78]. With the ability to observe surface and microstructures in detail, SEM is becoming a critical tool in developing and optimizing cellulose fiber-based mortars for construction applications requiring high durability.



(a) (b)

(c) (d)

Fig. 7. (a) SEM mixed mortar (N) at 1000x, (b) SEM mixed mortar (CF) at 1000x., (c) SEM of mortar mixture (NC) at 1000x. and, (d) SEM mixed mortar (CA) at 1000x

Fig. 7(c) SEM testing on nanocellulose (NC) based mortars provides a deeper understanding of the microstructure and material interactions at the nano level. Nanocellulose, which is obtained from biomass such as wood or agricultural waste, is recognized as a potential reinforcing material in mortar matrices. The use of SEM allows observation of the surface morphology and distribution of dispersed nanocellulose fibers in the cement matrix [79]. With high resolution, SEM can reveal microscopic structures, such as the thickness of the nanocellulose layer, the interaction between particles and the cement matrix, and the porosity formed in the mortar. Research by [80] found that the addition of nanocellulose can increase tensile strength and resistance to crack propagation, which is reflected in a smoother, well-structured surface morphology. SEM also helps in analyzing the dispersion of nanocellulose in cement, thereby reducing the inconsistency of particle distribution and improving cohesion between materials. In addition, SEM is used to evaluate changes in the microstructure of mortars due to variations in the water-cement ratio and compaction time.

Fig. 7(d) SEM testing on cellulose ash (CA) mortar provides an in-depth picture of the microstructure and properties of the material at the nano-level. Cellulose ash, resulting from the combustion of cellulose materials, is widely used as a partial substitute for cement in mortar composites to improve the material's physical and mechanical properties. SEM is used to examine the surface morphology of mortars containing cellulose ash, revealing details of ash particle distribution, interactions between cellulose ash and other components such as cement, aggregate, and water, as well as changes in microstructure during compaction and hardening [81].

The high resolution of SEM allows the observation of surface textures and micro-pores that can affect the strength and resistance of the mortar to external factors such as moisture and freeze-thaw [82]. Research by [83] reported that the use of cellulose ash can improve the porosity and water absorption ability of mortars, as seen from the changes in the microstructure of the SEM image. SEM also helps evaluate the influence of variations in the proportion of cellulose ash on the microstructure of mortars, including the distribution of particles and the formation of microcracks on the material's surface [84]. In addition, SEM analysis allows the identification of potential inconsistencies in mixing and compaction that can affect final performance, such as corrosion resistance and freeze-thaw cycles [85]. With SEM applications, researchers can optimize the use of cellulose ash in mortar to create materials with better durability in various construction conditions [86]. As such, SEM testing has become crucial in the analysis and development of modern mortars, as it provides a detailed picture of the relationship between microstructures and material performance at various scales of construction applications.

3.6. Energy Dispersive X-Ray (EDX)

Analyzing chemical elements in construction materials, such as mortar, is essential for understanding changes in their physical, chemical, and mechanical properties due to the addition of both organic and inorganic additives. One of the widely used methods for this purpose is Energy Dispersive X-ray Spectroscopy (EDX), which is usually integrated with the Scanning Electron Microscope (SEM) tool. Through this technique, researchers can identify and measure the content of major and minor elements in a sample, as well as map the elemental distribution microscopically. This approach is essential in the development of environmentally friendly material-based mortars, as it can help design optimal compositions that support technical performance and environmental sustainability.

EDX works by utilizing the interaction between X-rays and atoms in the sample. When a high-energy electron hits the surface of a sample, electrons from the skin of the atom are released. This electron vacuum is then filled by electrons from the outer shell, and the excess energy is released in the form of X-rays of specific energy. Each element has a unique X-ray energy spectrum, so EDX can be used to identify and calculate the relative content of each element present in the sample. By analyzing the energy peaks in the produced EDX spectrum, we can determine what elements are present in the mortar, their quantities, and how they are distributed in the material's microstructure [87].

Fig. 8(a) Normal mortar without additives (N), EDX results show that calcium (Ca) is the dominant element after oxygen (O). The highest energy peak on the EDX spectrum is located at around 3.7 keV, which corresponds to the characteristic energy of calcium. The high content of Ca (16.5%) is supported by very abundant oxygen (64.3%), reflecting the dominance of cement hydration compounds such as portlandite ($Ca(OH)_2$) and C-S-H (calcium silicate hydrate) gel in the mortar structure. In addition, the element carbon (C) is present in moderate levels (10.3%), while silica (Si), aluminum (Al), sodium (Na), magnesium (Mg), sulfur (S), and potassium (K) are found in smaller amounts. The EDX spectrum also shows that there are no significant peaks above four keV, indicating that other heavy elements, such as iron (Fe), are almost undetectable in normal mortars [88]. In general, this type of chemical composition is a key characteristic of Portland cement-based mortars, where calcium is the main element contributing to the compressive strength and stability of the hydration phases [89].

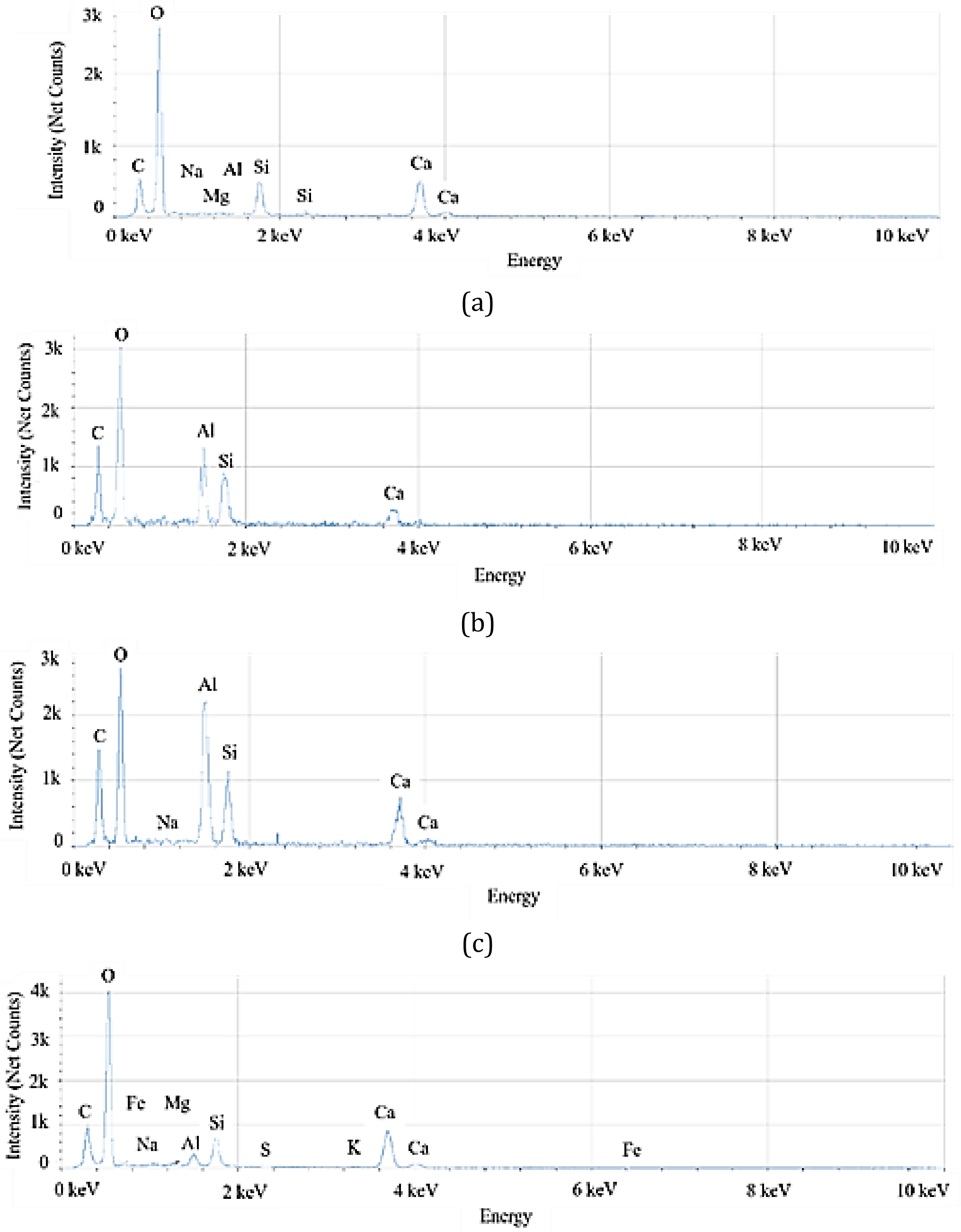
Fig. 8(b) The cellulose fibers from the waste paper are added to the mortar (CF), and the chemical composition undergoes quite noticeable changes. In the EDX spectrum, the highest peaks shift to lower energies, particularly around 0.5 keV, indicating oxygen [90]. The oxygen content (50.2%) remains high but relatively lower than normal mortar. Uniquely, the carbon content jumped significantly to 22.7%, almost double that of standard mortar. This increase in carbon clearly impacts the influx of organic fibers that are chemically rich in carbon. In addition, there was a substantial increase in aluminum (Al), reaching 9.7%, and silica (Si), reaching 8.8%. The increase in Al and Si may be due to an interaction between organic compounds in the fiber and the cement hydration product, resulting in complex tissues in the mortar matrix. The calcium content decreased drastically to 7.6%, which indirectly implicated changes in the hydration phase and compressive strength. While other elements such as Na, Mg, and K remain at very low or undetectable levels, this signifies that the addition of cellulose fibers does not significantly affect the alkaline elements in the system [91]. The absence of significant peaks above four keV, along with the undetectability of iron, supports the conclusion that light elements still dominate this mortar system.

Fig. 8(c) For nanocellulose (NC) based mortars, the EDX results show a slightly different chemical composition. The highest peak on the EDX spectrum shifted to an energy of about 1.7 keV, indicating the dominance of silica (Si) in the mortar matrix. The Si content (11.7%) was the highest among all mortar variations, indicating that nanocellulose, although organic-based, is likely to enhance pozzolanic reactions or improve the dispersion of silica aggregates in the matrix. Calcium (Ca) also experienced a significant increase of up to 18.3%, which is higher than in normal mortar. This suggests that nanocellulose can function as a nucleating agent that accelerates the process of cement hydration and the formation of calcium silicate hydrate phases. Oxygen remained dominant (57.9%), followed by carbon (9.1%), which decreased relatively compared to CF mortar. The decrease in carbon in NC, compared to CF, can be explained by the smaller particle size, which makes it more easily dispersed and chemically bound in the cement matrix, resulting in little free carbon remaining. Aluminum and sodium were also detected in small amounts (Al 1.5%; Na is 0.8%), while Mg, K, S, and Fe are at very low or undetectable levels. The high Si/Ca ratio in NC mortar indicates an increase in the C-S-H gel ratio, which could strengthen the microstructure of the mortar and increase resistance to external chemical attacks [92].

Fig. 8(d) Cellulose ash (CA) also results in unique changes in the chemical composition of the mortar. In the EDX spectrum, the highest peak returns to around 3.7 keV, indicating a predominance of calcium (Ca) that reaches 16.2%. Oxygen also remained high (63.7%), supporting the existence of the primary hydration phase. However, in CA, the carbon (11.9%) is slightly increased compared to normal mortar, indicating organic residues that have not been fully decomposed during the cellulose combustion process. Silica in CA decreased drastically to 4.7%, while aluminum was also at a low level (1.6%). Uniquely, in CA, sodium and magnesium are found in tiny amounts (0.3% each), and potassium and sulfur are also present in very minimal amounts.

Interestingly, there is a small peak of around 6.4 keV, indicating the presence of 0.8% iron (Fe), which may have originated from the burning of cellulose waste or contamination from milling equipment [93]. Although small, the iron content can affect the chemical properties and resistance of mortars, especially in aggressive environmental applications. The scientific interpretation of the above chemical composition data can be expanded by comparing the ratios between key elements, e.g., the ratio of Si/Ca and Si/Al. In normal mortar (N), the ratio of Si/Ca is 5.5/16.5 ≈ 0.33, and the ratio of Si/Al is 5.5/1.9 ≈ 2.89. This ratio shows the dominance of the calcium phase in conventional mortars [94]. In CF mortars, the Si/Ca ratio increases to 8.8/7.6 ≈ 1.16, and Si/Al to 8.8/9.7 ≈ 0.91, which signifies proportions of aluminum and silica that are almost equivalent to calcium, so that it may form a more complex secondary hydration network. For NC mortars, the Si/Ca ratio is 11.7/18.3 ≈ 0.64, and the Si/Al is 11.7/1.5 ≈ 7.8, showing a significant improvement in the ratio of Si/Al and Si/Ca that can support the formation of a more stable and stronger C-S-H gel. In CA mortars, the Si/Ca ratio was 4.7/16.2 ≈ 0.29, and Si/Al was 4.7/1.6 ≈ 2.94, again showing the dominance of calcium with a decrease in silica and aluminum content.

From a material perspective, changes in chemical composition due to the addition of cellulose-based additives have an impact on the physical and mechanical properties of mortar. An increase in the carbon content of CF mortar indicates a good distribution of fibers. However, if the amount is too small, it can decrease the compressive strength due to a decrease in the Ca phase content and the possibility of voids appearing in the matrix. In contrast, in NC mortars, increased calcium and silica, along with a higher Si/Ca ratio, indicate the formation of denser and stronger microstructures. The results of mechanical and SEM tests show densification and decreased porosity in nanocellulose-based mortars. CA mortar with cellulose ash exhibits similar behavior to normal mortar, but with the addition of the element Fe and a slight increase in carbon. This can improve or even decrease the resistance properties of the material, depending on the proportion and distribution of the element in the mortar matrix.



(d)

Fig. 8. (a) EDX mortar mixture (N)., (b) EDX mixed mortar (CF)., (c) EDX mixed mortar (NC)., (d) EDX mixed mortar (CA)

The presence of other elements, such as aluminum, sodium, magnesium, and sulfur, in low levels can act as impurities or minor phase formations, affecting resistance to chemical reactions, corrosion processes, or sulfate attacks. In compositions with a high Si/Ca ratio, such as in NC mortars, the possibility of C-S-H gel formation is greater, which can increase the compressive strength, flexibility, and durability of the mortar against aggressive environments such as freezing-thaw cycles or chloride ion exposure. Likewise, a high Si/Al ratio can help improve hydration phase stability at high temperatures or in extreme chemical environments. EDX can also be used to assess the dispersion and homogeneity effects of additives in mortar matrices. In NC mortars, the decrease in carbon compared to CF indicates a better distribution of nanocellulose, so it is not detected as much as free carbon. This is also supported by SEM observations that reveal nanofiber networks that are well integrated into the C-S-H gel. In addition, in CA mortars, the detection of iron, even in small amounts, can be interpreted as a by-product of the combustion or contamination process. In some cases, this can trigger secondary reactions, such as the formation of iron oxide, which is inert or even stabilizes the cement matrix. EDX analysis of various mortar mixtures provides an in-depth picture of the chemical composition changes that occur due to the addition of cellulose-based additives. Each variation of the additive gives rise to a distinctive pattern of elemental distribution, which is closely correlated with changes in the microstructural and mechanical properties of the mortar. EDX data enabled researchers to design innovative mortar formulas that are not only mechanically strong but also environmentally friendly and resistant to real-world conditions and challenges in the field. These results are also an important basis for the development of future construction materials that prioritize resource efficiency and environmental sustainability. The value of the chemical composition in the EDX test can be seen in Table 2.

Table 2. Chemical composition of cellulose mortar

Chemical Composition	Percentage (%)			
	N	CF	NC	CA
C	10,3	22,7	9,1	11,9
O	64,3	50,2	57,9	63,7
Na	0,4	0,5	0,8	0,3
Mg	0,3	0,5	0,2	0,3
Al	1,9	9,7	1,5	1,6
Si	5,5	8,8	11,7	4,7
S	0,4	-	0,5	0,3
K	0,4	-	-	0,2
Ca	16,5	7,6	18,3	16,2
Fe	-	-	-	0,8

4. Conclusion

This study highlights the potential of nanocellulose synthesized from waste paper as a sustainable additive to enhance mortar's microstructural and mechanical performance. Utilizing waste paper as a raw material provides a valuable route for waste valorization and contributes to the circular economy and sustainable construction practices. The low-temperature synthesis process adopted in this study demonstrated higher energy efficiency and lower carbon emissions than conventional high-temperature approaches, confirming its environmental advantages.

Incorporating nanocellulose at a dosage of 2% by weight of cement led to notable improvements in mortar performance. Compressive strength increased by approximately 25%, from 28.72 MPa for the control mix to 30.16 MPa for the nanocellulose-modified mortar. These results indicate that nanocellulose enhances the interfacial bonding within the cement matrix, leading to a denser microstructure and improved stress transfer. Scanning Electron Microscopy (SEM) analysis further confirmed a uniform dispersion of nanocellulose fibers, reduced micro voids, and enhanced cohesion among hydration products, contributing to better structural integrity. Flow testing showed a minor reduction in workability, with a flow diameter of 152 mm compared to 165 mm for the control mortar; however, the value remained within an acceptable range for practical construction use. Porosity analysis revealed a 15% reduction in pore volume, indicating enhanced resistance to water ingress and environmental degradation. These findings demonstrate that nanocellulose can effectively refine the pore structure and increase durability without compromising workability.

From an environmental standpoint, producing nanocellulose from waste paper reduced the carbon footprint by up to 30% compared with production from virgin cellulose sources, reinforcing its potential as a green material for construction applications. While scalability and production cost remain challenges, advancements in synthesis and processing technologies are expected to improve feasibility. Overall, nanocellulose derived from waste paper presents a promising, eco-friendly solution for developing next-generation sustainable building materials.

Acknowledgement

We express our deepest gratitude to all lecturers at the Doctoral Study Program in Civil Engineering, Faculty of Engineering, Sriwijaya University, and all parties involved in this research, directly and indirectly. The support provided is significant so that this research can be completed satisfactorily. We hope that the results of this research can make a valuable contribution to the development of science and positively impact the progress of the construction industry.

References

[1] Yuxuan C, Longfei Z, Kai W, Huaxing G, Zhengyao L, Yuanshan S, Feixiang C, Rheology control and shrinkage mitigation of 3D printed geopolymer concrete using nanocellulose and magnesium oxide. Construction and Building Materials, 2024; 429: 136421. https://doi.org/10.1016/j.conbuildmat.2024.136421

[2] Muhammad N, Muhammad AA, Mukarram Z, Noman A, Tag NH, Moath KA, Mohammad SM, Wal A, Mamdouh AA. Engineered cellulose nanocrystals-based cement mortar from office paper waste: Flow, strength, microstructure, and thermal properties. Construction and Building Materials, 2024; 417: 134123.

[3] Pengki S, Saloma, Siti NA, Shek PN. Synthesis of nanocellulose as a sustainable construction material from waste paper using the alkaline method at low temperature. Buildings, 2024; 14(5): 1298.

[4] Pengki S, Saloma, Arie PA, Anis A, Mohammad I, Nur HAK. The Characterization of Nanocellulose with Various Durations and NaOH Concentration. International Journal of Innovatie Research and scientific Studied, 2022; vol. 5, no. 1, pp. 18-29. https://doi.org/10.53894/ijirss.v5i1.343

[5] Malgorzata S, Ewelina GB, Dauta BN, Eric Nl. Critical Review on Modification Methods of Cement Composites with Nanocellulose and Reaction Conditions during Nanocellulose Production. Materials, 2022 vol. 15, no. 21, p. 7706. https://doi.org/10.3390/ma15217706

[6] Taiwo A, P.S Mangat. Physical, Mechanical and Chemical Properties of nanocellulose Fibre Reinforced Mortar. Sheffield Hallam University Research Archive, 2024.

[7] Do YH, Zhen LL, Darren YSL, Siah YT, Sivakumar M, Khang WT, Zhen HB. .Ultrasonic cavitation: An effective cleaner and greener intensification technology in the extraction and surface modification of nanocellulose, Ultrasonic Sonochemistry, 2022, vol. 90, p. 106176. https://doi.org/10.1016/j.ultsonch.2022.106176

[8] Seth K, Sabbie AM, Kimberly EK, Jeffrey PY, Eric NL, W. Jason W. Harmonized Life-Cycle Inventories of Nanocellulose and Its Application in Composites. Environmental Science & Technology, 2023, vol. 57, no. 48, pp. 19137-19147. https://doi.org/10.1021/acs.est.3c04814

[9] Sevilay D, Algan T. Investigating major challenges for industry 4.0 adoption among construction companies. Enggineering Construction Architectural Managemen, 2021, 29 (3): 1470-1503

[10] Junlei X, Huiling L, Hua Z, Shujian H, Qian Z, Kunming L, Shaohua J, Gaigai D, Kai Z. Nanocellulose and its derived composite electrodes toward supercapacitors: Fabrication, properties, and challenges, Journal of Bioresources and Bioproducts, 2022, vol. 7, no. 4, pp. 245-269. https://doi.org/10.1016/j.jobab.2022.05.003

[11] Zehong C, Yijie H, Ge S, Hao Z, Mohammed AA, Ewelina J, Huili z, Linxin Zhong, Xinwen P. Advanced Flexible Materials from Nanocellulose. Advanced Functional Materials, 2023, vol. 33, no. 18. https://doi.org/10.1002/adfm.202214245

[12] Xinya M, Youzhu L, Xiaojin G, Ming S. Improvement of ultra-high-performance concrete matrix with nanocellulose for reduced autogenous shrinkage and enhanced mechanical properties. Construction and Building Materials, 2024, vol. 457, p. 139418. https://doi.org/10.1016/j.conbuildmat.2024.139418

[13] Perry LNK, Md Rezaur R, Muhammad KBB. A review on the extraction of cellulose and nanocellulose as a filler through solid waste management. Journal of Thermoplastic Composite Materials, 2021, vol. 36, no. 3, pp. 1306-1327. https://doi.org/10.1177/08927057211020800

[14] Aiai W, Shuai C, Erol Y. Influence of types and contents of nano cellulose materials as reinforcement on stability performance of cementitious tailings backfill. Construction Building Materials, 2022, vol. 344, p. 128179. https://doi.org/10.1016/j.conbuildmat.2022.128179

[15] Zhanghui W, Zhuo C, Zhaodi Z, Hongzhi L, Liping Z, Mingcheng Y, Yang C. Nanocellulose-based membranes for highly efficient molecular separation. Chemical Engineering Journal, 2023, vol. 451, p. 138711. https://doi.org/10.1016/j.cej.2022.138711

[16] Muhammad AA, Mukarram Z, Khalid SA, Muhammad N, Mohammad SM, Tag NH, Yasir MA. Development and Evaluation of Engineered Nanocellulose-Based Mortar Synthesized Using Hydrolysis of Strong and Weak Acids. Arabian Journal for Science and Engineering, 2023, vol. 48, no. 10, pp. 13605-13614. https://doi.org/10.1007/s13369-023-07869-8

[17] Tobias B, Nico K, Malin N, Andreas BF, Gustav N, Lars W. The Colloidal Properties of Nanocellulose. ChemSusChem, 2023, vol. 16, no. 8. https://doi.org/10.1002/cssc.202201955

[18] Theresa K, Alexander B, Werner Z, Kathrin W, Alexander B, Andreas M, Francisco M, Andreas G, Iris K. Environmental life cycle assessment of nano-cellulose and biogas production from manure. Journal of Environmental Management, 2022, vol. 314, p. 115093. https://doi.org/10.1016/j.jenvman.2022.115093

[19] Warathorn C, Jidapha T. Nanocellulose production from rice straw derived-cellulose by enzymatic hydrolysis and its effect on lipase activity. Asia-Pacific Journal of Science and Technology, 2022, vol. 27, no. 2, pp. 1-9.

[20] Peng W, Changyang L, dun Z. Recent advances in chemical durability and mechanical stability of superhydrophobic materials: Multi-strategy design and strengthening. Journal of Materials Science & Technology, 2022, vol. 129, pp. 40-69. https://doi.org/10.1016/j.jmst.2022.01.045

[21] Tuhua Z, Guoqing J, zhen C, Michael W, somayeh N, Carlos AF.Interfacial interactions and reinforcing mechanisms of cellulose and chitin nanomaterials and starch derivatives for cement and concrete strength and durability enhancement: A review. Nanotechnology Reviews, 2022, vol. 11, no. 1, pp. 2673-2713. https://doi.org/10.1515/ntrev-2022-0149

[22] E.G. Deze, E. Cuenca, A.M.L Nasner, M. Iakovlev, A. Sideri, A. Sapalidis, R.P. Borg, L. Ferrara. Nanocellulose enriched mortars: Evaluation of nanocellulose properties affecting microstructure, strength and development of mixing protocols. Materialstoday: Proceedings, 2022. https://doi.org/10.1016/j.matpr.2021.09.511

[23] Laleh s, Valentina G, Katja H, Lina S, Elina N, Wenyang X, Karl Mihhels, Marcel Kroger, Zhoujun M, Jakob W, Han T, Emily DC, Eeero K. Understanding Nanocellulose-Water Interactions: Turning a Detriment into an Asset. Chemical Review, 2023, vol. 123, no. 5, pp. 1925-2015. https://doi.org/10.1021/acs.chemrev.2c00611

[24] Ana IM, Joao M, Paulo M, Maria DRV, Carlos S, Paulo C, Joao G, Ferreira. Modulus of elasticity of mortars: Static and dynamic analyses. Construction and Building Materials, 2019, vol. 232, p. 117216. https://doi.org/10.1016/j.conbuildmat.2019.117216

[25] Guancheng J, Jinsheng S, Yinbo H, Kaixiao C, Tengfei D, Lili Y, Xukun y, Xingxing W. Novel Water-Based Drilling and Completion Fluid Technology to Improve Wellbore Quality During Drilling and Protect Unconventional Reservoirs. Engineering, 2022, vol. 18, pp. 129-142. https://doi.org/10.1016/j.eng.2021.11.014

[26] Javad S, Ali RTK, Mohammad C, Ali K. Effects of particle gradations on cyclic behavior of ballast contaminated with sand. Construction and Building Materials, 2022, vol. 342, p. 127943. https://doi.org/10.1016/j.conbuildmat.2022.127943

[27] Boweng f, Jiesheng L, Zhenzhen L, Man Z, Xiaoming T. Study on properties and durability of alkali activated rice straw fibers cement composites. Journal of Building Engineering, 2023, vol. 63, p. 105515. https://doi.org/10.1016/j.jobe.2022.105515

[28] Alexander L, Yu KT, Chi-hwa W, Harn WK, Hayden T. Utilization of waste materials in a novel mortar-polymer laminar composite to be applied in construction 3D-printing. Composite structure, 2020, vol. 253, p. 112764. https://doi.org/10.1016/j.compstruct.2020.112764

[29] Yiming L, Haibo D, Yihua N, Simon AMH. Effective control of flexible asphalt pavement cracking through quality assurance testing of extracted and recovered binders. Construction and Building Materials, 2021, vol. 273, p. 121769. https://doi.org/10.1016/j.conbuildmat.2020.121769

[30] Eric S, Gotz H, Anthony J, Alexander M, Christoph S. Calculating Rheological Properties of Fresh Mortar for Additive Manufacturing Based on Experimental, Multi-Sensor Data. Advances in science and technology, 2024, pp. 131-139. https://doi.org/10.4028/p-EV4gPv

[31] Christian O, Sandhya B, Somnuk T, Takafumi S. Comparison of environmental impacts of fly ash and slag as cement replacement materials for mass concrete and the impact of transportation. Sustainable Materials and technologies, 2024, vol. 39, p. e00796. https://doi.org/10.1016/j.susmat.2023.e00796

[32] John J, Fernando F, Braden D, Michael R, Thodore M. Effect of size and water content on the compressive strength of mortar. Construction and Building Materials, 2023, vol. 368, p. 130505. https://doi.org/10.1016/j.conbuildmat.2023.130505

[33] Mohammad KA, Mustafa SN, Sagheer AO, Ibnelwaleed AH, Mohammad KH. Production of high-performance concrete through the addition of new generation of superplasticizers: a comprehensive review on the electrokinetics and rheological behaviors. Emergent Materials, 2023, vol. 7, no. 2, pp. 403-441. https://doi.org/10.1007/s42247-023-00550-8

[34] Branavan A, Chaminda K, Anura N. Performance evaluation of cement mortar produced with manufactured sand and offshore sand as alternatives for river sand.Construction and building materials, 2021, vol. 297, p. 123784. https://doi.org/10.1016/j.conbuildmat.2021.123784

[35] Jun L, Luxi Z, Lin C, Guo L, Tan L, Shenyu C. Effect of multilayer graphene oxide on the hydration and early mechanical strength of cement mortar in low temperature. Construction and building materials, 2023, vol. 364, p. 129997. https://doi.org/10.1016/j.conbuildmat.2022.129997

[36] Mohd J, Abid H, Ravi PS, Rajiv S, Shanay R. Role of additive manufacturing applications towards environmental sustainability. Advanced Industrial and Engineering Polymer Research, 2021, vol. 4, no. 4, pp. 312-322. https://doi.org/10.1016/j.aiepr.2021.07.005

[37] Gioele V, Julien B, Max F, Serge R. From fundamentals in calibration to modern methodologies: A tutorial for small molecules quantification in liquid chromatography-mass spectrometry bioanalysis. Analytica Chimica Acta, 2023, vol. 1240, p. 340711. https://doi.org/10.1016/j.aca.2022.340711

[38] Florentina V, Milagros R, Aime R, John S, Marta FG. Sampling and analysis techniques for inorganic air pollutants in indoor air. Applied Spectroscopy Reviews, 2022, vol. 57, no. 7, pp. 531-579. https://doi.org/10.1080/05704928.2021.2020807

[39] Alexandra VB, Iulian B. Chitosan-Based Electrochemical Sensors for Pharmaceuticals and Clinical Applications. Polymers, 2023, vol. 15, no. 17, p. 3539. https://doi.org/10.3390/polym15173539

[40] Ahmed F, Diana P, Vitor HC, Susana OC, Graca M. Fourier Transform Infrared (FTIR) Spectroscopy to Analyse Human Blood over the Last 20 Years: A Review towards Lab-on-a-Chip Devices. Micromachines, 2022, vol. 13, no. 2, p. 187. https://doi.org/10.3390/mi13020187

[41] Guan LL, Sergei GK. Recent advances and applications to cultural heritage using ATR-FTIR spectroscopy and ATR-FTIR spectroscopic imaging. Analyst, 2022; 147(9), pp. 1777-1797. https://doi.org/10.1039/D2AN00005A

[42] Chayanika R, Mahua S, Priyansha G, Manish K, Akshata N, Jacob DB. Standardization of micro-FTIR methods and applicability for the detection and identification of microplastics in environmental matrices. Science of the environment, 2023, vol. 888, p. 164157. https://doi.org/10.1016/j.scitotenv.2023.164157

[43] Hossein O, sumana DC, Arnavaz A. Hydrogels in biosensing and medical diagnostics. Journal of Bioactive and Compatible Polymers, 2024, vol. 39, no. 6, pp. 480-506. https://doi.org/10.1177/08839115241253260

[44] Sylwia PP, Marcin C, Jolanta F. Application of Fourier Transform Infrared (FTIR) Spectroscopy in Characterization of Green Synthesized Nanoparticles. Molecules, 2025, vol. 30, no. 3, p. 684. https://doi.org/10.3390/molecules30030684

[45] Claudia C, Ilaria S, Carmine M, Vito FU. Fourier Transform Infrared Spectroscopy to Assess the Degree of Alteration of Artificially Aged and Environmentally Weathered Microplastics. Polymers, 2023, vol. 15, no. 4, p. 911. https://doi.org/10.3390/polym15040911

[46] V. K. Ahluwalia. Introduction to Instrumental Methods of Chemical Analysis. Instrumental Methods of Chemical Analysis, Cham: Springer Nature Switzerland, 2023, pp. 3-10. https://doi.org/10.1007/978-3-031-38355-7_1

[47] Amulya L, Bhisham s, Mary D, Denver S. Sound absorption and transmission loss properties of open-celled aluminum foams with stepwise relative density gradients. Applied Acoustics, 2022, vol. 193, p. 108780. https://doi.org/10.1016/j.apacoust.2022.108780

[48] Taiwo A, P.S. Mangat. Hydration Products and Properties of Nanocellulose Fibre-Reinforced Mortar. Sustainability, 2025, vol. 17, no. 6, p. 2719. https://doi.org/10.3390/su17062719

[49] Nandkishore T, Arti M, Deodhari s, Arnab RC, Mohammad FA. Comparative FTIR Characterization of Various Natural Gums: A Criterion for Their Identification. Journal of Polymers and the Environment, 2023, vol. 31, no. 8, pp. 3372-3380. https://doi.org/10.1007/s10924-023-02821-1

[50] Peeyush P, Shreya S, Ranjana J, Sukhvir S. Diffusion Controlled Features of Microwave Assisted ZnS/ZnO Nanocomposite with Reduced Band Gap. ECS Journal of Solid State Science and Technology, 2023, vol. 12, no. 3, p. 034004. https://doi.org/10.1149/2162-8777/acc426

[51] Shajahan A, R. Shenbagaraman. Fourier transform infrared spectroscopy in characterization of bionanocomposites. Characterization Techniques in Bionanocomposites, 2025, pp. 209-227. https://doi.org/10.1016/B978-0-443-22067-8.00008-3

[52] Asif A, Haris A. Fourier Transform Infrared Spectroscopy (FTIR) Technique for Food Analysis and Authentication. Nondestructive Quality Assessment Techniques for Fresh Fruits and Vegetables, Singapore: Springer Nature Singapore, 2022, pp. 103-142. https://doi.org/10.1007/978-981-19-5422-1_6

[53] Md. Mahmudur R, Md. Aftab AS, Mst. Sarmina Y, Md. Abdul G. Md. Ismail H, Md. Ashraful A. Md. Salim K. Trisha P, Md. Saiful Q. Simultaneous removal of Ni2+ and Congo red from wastewater by crystalline nanocellulose - Modified coal bionanocomposites: Continuous adsorption study with mathematical modeling. Groundwater for Sustainable Development, 2024, vol. 26, p. 101244. https://doi.org/10.1016/j.gsd.2024.101244

[54] Soumaya Z, Fadhel A, Habib S. Effects of Cationic Surfactant on Fresh and Hardened Properties of Cement-Based Mortar. Journal of Renewable Materials, 2023, vol. 11, no. 5, pp. 2345-2365. https://doi.org/10.32604/jrm.2023.026170

[55] S. Sahila, L. S. Jayakumari, P. S. Sampath, S. Palanisamy, and M. Palaniappan. Cellulose Based Rubber Blends and Microcomposites. Rubber based bionanocomposites, 2024, pp. 45-73. https://doi.org/10.1007/978-981-10-2978-3_3

[56] Sheng H, Yuxi L, Peng Y, Yitong Z. Effect of lime mud under wet grinding on the compressive strength and hydration of cement mortar. Cement and concrete composites, 2023, vol. 140, p. 105067. https://doi.org/10.1016/j.cemconcomp.2023.105067

[57] Jiayu L, Shengqian R, Yi L, Tao w, Qiang Z, Dongming Y. Morphological characteristics of calcium carbonate crystallization in CO 2 pre-cured aerated concrete. RSC Advances, 2022, vol. 12, no. 23, pp. 14610-14620. https://doi.org/10.1039/D2RA01901A

[58] Moruf OY. Bond Characterization in Cementitious Material Binders Using Fourier-Transform Infrared Spectroscopy. Applied sciences, 2023, vol. 13, no. 5, p. 3353. https://doi.org/10.3390/app13053353

[59] Leticia ODS, Marco L, Lourdes MSDS, Flavio DAS, Viktor M. Effect of microcrystalline and nano-fibrillated cellulose on the mechanical behavior and microstructure of cement pastes. Construction and Building Materials, 2023, vol. 408, p. 133812. https://doi.org/10.1016/j.conbuildmat.2023.133812

[60] Tzu-Yi Y, Yu-Kai T, Ting-Han L, Tzu-Chia W, Yun-Hsiu T, Yin-Hsuang C, Ming-Chung W, Wei-Fang S. Effect of cellulose compositions and fabrication methods on mechanical properties of polyurethane-cellulose composites. Carbohydrate Polymers, 2022, vol. 291, p. 119549. https://doi.org/10.1016/j.carbpol.2022.119549

[61] Obinna O, Nemkumar B. Sulfate resistance of cement composites containing Nano-Fibrillated Cellulose (NFC). Cement and Concrete Composites, 2023, vol. 135, p. 104831. https://doi.org/10.1016/j.cemconcomp.2022.104831

[62] Maria VD, Cristina T, Mariagiovanna T, Giangiacomo T, Nicolò M, Chiara T, Elena V. Suspended Multifunctional Nanocellulose as Additive for Mortars. Nanomaterials, 2022, vol. 12, no. 7. https://doi.org/10.3390/nano12071093

[63] Muhammad IH, Warda A, Rakibul IK, Surendra S. A comparative investigation on the effects of nanocellulose from bacteria and plant-based sources for cementitious composites. Cement and Concrete Composites, 2022, vol. 125, p. 104316. https://doi.org/10.1016/j.cemconcomp.2021.104316

[64] Bai Z, Hong Z, Pan F, Pu Z. A review on shrinkage-reducing methods and mechanisms of alkali-activated/geopolymer systems: Effects of chemical additives. Journal of Building Engineering, 2022, vol. 49, p. 104056. https://doi.org/10.1016/j.jobe.2022.104056

[65] Yidong G, Hongzhi Z, Minfei L, Yu Z, Erik S, Klaas VB, Branko S. Flexural strength and fatigue properties of interfacial transition zone at the microscale. Cement and Concrete Composites, 2022, vol. 133, p. 104717. https://doi.org/10.1016/j.cemconcomp.2022.104717

[66] Long L, Xin Z, Qiaoling L, Xiurong L, Xingyan S. Cellulose nanofibrils for the performance improvement of ultra-high ductility cementitious composites. Cellulose, 2022, vol. 29, no. 3, pp. 1705-1725. https://doi.org/10.1007/s10570-022-04421-z

[67] Simone D, Aurelio B, Alex D, Antonia N. Lattice Strain and Defects Analysis in Nanostructured Semiconductor Materials and Devices by High-Resolution X-Ray Diffraction: Theoretical and Practical Aspects. Small Methods, 2022, vol. 6, no. 2. https://doi.org/10.1002/smtd.202100932

[68] Xin L, Si-wei M, Zheng-zhou L, Chun'an T, Jia-Ping T, Ji-Zhou T. Microscale crack propagation in shale samples using focused ion beam scanning electron microscopy and three-dimensional numerical modeling. Petroleum Science, 2023, vol. 20, no. 3, pp. 1488-1512. https://doi.org/10.1016/j.petsci.2022.10.004

[69] Zhao-Qi L, Hongyun C, Jie D, Xin Y, Shi-rong Z, Ying-Hao Z, Yang L. Study of the Failure Mechanism of Mortar Rubble Using Digital Image Correlation, Acoustic Emission and Scanning Electron Microscopy. Buildings, 2022, vol. 12, no. 9, p. 1313. https://doi.org/10.3390/buildings12091313

[70] Mostafa A, Fatemaeh P. Evaluating the performance of Self-Sensing concrete sensors under temperature and moisture variations- a review. Construction and Building Materials, 2023, vol. 404, p. 132923. https://doi.org/10.1016/j.conbuildmat.2023.132923

[71] Boyuan Y, S. Kamali-Bernard. F. Bernard. Microstructure, tensile strength and shear strength of aggregate-mortar interface: Effect of aggregate mineralogy. Construction and Building Materials, 2023, vol. 388, p. 131721. https://doi.org/10.1016/j.conbuildmat.2023.131721

[72] Xin S, Ping Y, Lin L, Xueyu G, Xin L, Jiling Z. Strength and microscopic pore structure characterization of cement-fly ash stabilized organic soil under freeze-thaw cycles. Construction and Building Materials, 2024, vol. 420, p. 135635. https://doi.org/10.1016/j.conbuildmat.2024.135635

[73] Runfang Z, Haitao M, Shuai Y, Zhengcheng W, Xiaobing H. Research on mechanical properties and pore structure evolution process of steam cured high strength concrete under freeze-thaw cycles. Scientific reports, 2024, vol. 14, no. 1, p. 27530. https://doi.org/10.1038/s41598-024-76631-3

[74] Emircan O, Anil K, Muhammed FG, Behlul FO, Gurkan Y, Ashraf A, Mustafa S. A comprehensive study on the compressive strength, durability-related parameters and microstructure of geopolymer mortars based on mixed construction and demolition waste. Journal of Cleaner Production, 2023, vol. 396, p. 136522. https://doi.org/10.1016/j.jclepro.2023.136522

[75] Sarah F, Chamila G, Amin S, Kate N, Massoud S, Sujeeva S, Priyan M, Md. Tareq R. Sustainable Cement Composite Integrating Waste Cellulose Fibre: A Comprehensive Review. Polymers, 2023, vol. 15, no. 3, p. 520. https://doi.org/10.3390/polym15030520

[76] Lin F, Xin Y, Chen C, Pingping H. Synergistic mechanism of polycarboxylate superplasticizer and hydroxypropyl methyl cellulose on fiber dispersion and mechanical properties of PVA-fiber concrete: experimental and simulation studies. Journal of Sustainable Cement-Based Materials, 2025, vol. 14, no. 5, pp. 983-1004. https://doi.org/10.1080/21650373.2025.2487889

[77] Yupeng L, Wei J, Jingjing X, Funan Z, Shuangiao Z, Chengwei X, Dongdong Y. Effects of kneading and impact action on the movement of aggregates in asphalt mixtures during compaction. Construction and Building Materials, 2023, vol. 366, p. 130210. https://doi.org/10.1016/j.conbuildmat.2022.130210

[78] Haolong G, Hailong W, Huijun X, Hao L, Yur L, Lisi W. Study on damage deterioration mechanism and service life prediction of hybrid fibre concrete under different salt freezing conditions. Construction and Building Materials, 2024, vol. 435, p. 136688. https://doi.org/10.1016/j.conbuildmat.2024.136688

[79] Parichat T, Kittisak J, Choncharoen S, Jonghwan S, Kittiphat K, Pitiwat W, Pornchai R. Microstructural Characterization of Cellulose Nanocrystals and Microcellulose from Bamboo (Bambusa longispatha) for Reinforcing Ordinary Portland Cement Matrix. Polymers, 2024, vol. 16, no. 24, p. 3558. https://doi.org/10.3390/polym16243558

[80] Sultan C, Harun A, Idris K. Mechanical, thermal, and morphological properties of nanocellulose reinforced ABS nanocomposites. Cellulose, 2024, vol. 31, no. 16, pp. 9715-9729. https://doi.org/10.1007/s10570-024-06193-0

[81] R. Sri BR, Pitabash S, Souradeep G. Effect of microcrystalline cellulose on rheology, hydration kinetics and early-age properties of Portland cement-based and alkali-activated slag-fly ash blend. Journal of Building Engineering, 2023, vol. 76, p. 107218. https://doi.org/10.1016/j.jobe.2023.107218

[82] Ghasan FH, Masoumeh K, Ziyad K, Omrane B, Mohammad J, Mahmoodi. Freeze-thaw cycle and abrasion resistance of alkali-activated FA and POFA-based mortars: Role of high volume GBFS incorporation. Heliyon, 2023, vol. 9, no. 7, p. e17672. https://doi.org/10.1016/j.heliyon.2023.e17672

[83] Mei-Yu X, Xiao-Yong W. Multi-technique investigation regarding the impact of cellulose nanofibers on ultra-high-performance concrete at the macroscopic and microscopic levels. Construction and Building Materials, 2022, vol. 327, p. 126936. https://doi.org/10.1016/j.conbuildmat.2022.126936

[84] Heba N, Ibrahim GA, Andrea D, Imre C, Mark H. Microstructural Characterisation of Adsorbent Ash with Potentially Toxic Elements in a Mortar. Ecological Chemistry and Engineering S, 2024, vol. 31, no. 1, pp. 103-116. https://doi.org/10.2478/eces-2024-0008

[85] Mohammadmahdi A, Mohammad JR, Mostafa A, Mohammed SM, Baogo H, Raul F, Antonio GC. Influence of cement and water content on the multifaceted capabilities of a self-sensing cement-based geocomposite: a comprehensive analysis. Measurement Science and Technology, 2024, vol. 35, no. 8, p. 085606. https://doi.org/10.1088/1361-6501/ad4c89

[86] Amirshosein SM, Alireza MGR, Fereydoon O. Utilization of agricultural wastes as fiber, binder and aggregates of geopolymer mortars: Application of taguchi method for strength and durability optimization. Journal of Building Engineering, 2023, vol. 75, p. 106906. https://doi.org/10.1016/j.jobe.2023.106906

[87] Junsheng L, Wenyuan R, Aijun Z, Shuangcun L, Jianping T, Hongtai L. Mechanical Properties and Microstructure Analysis of Cement Mortar Mixed with Iron Ore Tailings. Buildings, 2023, vol. 13, no. 1, p. 149. https://doi.org/10.3390/buildings13010149

[88] Laiming H, Yi W, Ming'an S. Micromorphology, mineralogy, and geochemistry of ferromanganese nodules in tropical soils and their impacts on heavy metal sequestration. Journal of Cleaner Production, 2023, vol. 383, p. 135498. https://doi.org/10.1016/j.jclepro.2022.135498

[89] Aso AA, Ahmed SM, Serwan R, Riyadh Noaman, Warzer SQ, Kawan G, Hind A, Raed F. Microstructure, chemical compositions, and soft computing models to evaluate the influence of silicon dioxide and calcium oxide on the compressive strength of cement mortar modified with cement kiln dust. Construction and Building Materials, 2022, vol. 341, p. 127668. https://doi.org/10.1016/j.conbuildmat.2022.127668

[90] Chiara M, Gregorio C, Xavier G, Vincent P, Virginie BP, Anna K, Sylvie, Johnny B, Angelina D, Camille R, Landrot, Ana RLR, Dario L, Bernanrd M, Jean-Noel S, Aleksandra I, Mirjana K, Olivier H, Corina B, Marina R, Nadia M. Revealing the adsorption mechanism of copper on hemp-based materials through EDX, nano-CT, XPS, FTIR, Raman, and XANES characterization techniques. Chemical Engineering Journal Advances, 2022, vol. 10, p. 100282. https://doi.org/10.1016/j.ceja.2022.100282

[91] Moises F, A.M. Moreno R, Raquel VDLV, Rosario G, Sagrario MR, Jaime M, Asier O. Advances in the understanding of alkaline waste materials as potential eco-pozzolans: Characterisation, reactivity and behaviour. Journal of Building Engineering, 2024, vol. 92, p. 109744. https://doi.org/10.1016/j.jobe.2024.109744

[92] Hongbo W, Mingsheng H, Jianjun C, Tao W, Yali C, Yongkun D. Study on the synergistic effects of OPC and silica fume on the mechanical and microstructural properties of geopolymer mortar. Construction and Building Materials, 2024, vol. 434, p. 136740. https://doi.org/10.1016/j.conbuildmat.2024.136740

[93] Mehran G, Navya PN, Katia A, ali Z, Peter CS, Elena PI, Benu A, Minoo Naebe, Suresh KB. Fluorescent Nanocarbons: From Synthesis and Structure to Cancer Imaging and Therapy. Advanced Materials, 2024, vol. 36, no. 19. https://doi.org/10.1002/adma.202312474

[94] H. Shoukry, Priyadharshini P, Aref A, Hussam A, Mohammed A. Hamdy AA. Performance of limestone-calcined clay cement mortar incorporating high volume ferrochrome waste slag aggregate. Construction and Building Materials, 2022, vol. 350, p. 128928. https://doi.org/10.1016/j.conbuildmat.2022.128928