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Nanotechnology-Enabled Innovations in Cement: Enhancing Durability, and Impact Load Resistance with Nanocellulose, Nano Silica, and Nano Clay Additives

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Abstract

This article addresses the urgent need for sustainable advancements in concrete materials within the cement industry, a sector that consumes vast amounts of nonrenewable resources and contributes approximately 5% of global CO_2 emissions. The study investigates the effectiveness of nano cellulose, nano silica, and nano clay as partial replacements to enhance concrete properties. By examining the influence of varying nanoparticle percentages on compressive strength, flexural strength, water absorption, impact resistance, and abrasion resistance, this research provides valuable insights into their performance. The study identifies an optimal weight ratio of 0.75% nano cellulose for achieving maximum compressive strength, alongside a combination of 1.25% nano silica and 0.75% nano cellulose for superior flexural strength. However, it is noted that excessive concentrations of nano cellulose may result in agglomeration and uneven dispersion of nanoparticles, adversely affecting concrete performance. The article underscores the challenges associated with incorporating larger quantities of nanoparticles and emphasizes the necessity for further research to improve dispersion techniques, ultimately enhancing concrete strength and durability. Additionally, a brief overview of the study's experimental methods is included to provide clarity on the research approach. In conclusion, this study highlights the promising potential of nano cellulose, nano silica, and nano clay as additives for enhancing concrete performance. By optimizing dosages and addressing dispersion challenges, these nanoparticles offer a viable path toward sustainable concrete production with improved mechanical properties and enhanced durability.

Keywords: nanocellulose; nano silica; nano clay; durability; impact load resistance.

1. Introduction

The cement industry produces over 4 billion metric tons of Portland cement annually, which significantly contributes to the consumption of nonrenewable resources and approximately 25% of global CO2 emissions. This situation exacerbates climate change and poses serious health risks due to associated pollution. As a result, there is an urgent need for high-performance, durable, and sustainable alternative concrete materials. To address these challenges, a variety of additives, including supplemental cementitious materials (SCMs) and nanomaterials, are being utilized [1]. The rising demand for sustainable, eco-friendly, and biodegradable concrete has led to the development of second-generation cellulosic technologies derived from nanoscale cellulose extraction. Industries increasingly advocate for disposable products and solutions made from sustainable, ecologically beneficial, and renewable resources. Natural cellulose-based materials, such as cotton, wood, and linen, have been employed in structural applications for centuries due to their low cost, biodegradability, non-toxicity, and commendable mechanical performance [2,3]. The integration of nanoscale materials holds promise for enhancing performance efficiency through cellulose technologies [4,5]. This organic resource is known to impart improved mechanical strength, durability, and versatility across various concrete applications. Synthesised nanocellulose can be utilized in producing fibers, hydrogels, aerogels, and transparent films that exhibit exceptional mechanical and thermal properties [6]. Research indicates that incorporating varying percentages of nanocellulose as a partial cement replacement can enhance compressive strength by up to 30% [7]. The inclusion of nanocellulose in cementitious mixtures has been shown to improve fracture energy, flexural strength, toughness, and overall durability by creating a more homogeneous and less porous material, thereby reducing sulfate intrusion [8,9]. Numerous studies have demonstrated that while the incorporation of cellulose nanofibrils (CNF) in concrete may decrease workability, it significantly increases both flexural and compressive strength, likely due to enhanced hydration and compaction within the cement paste microstructure [4,10].A primary challenge associated with the incorporation of nanocellulose into the concrete matrix is its reduced durability when added in large quantities, primarily due to the excess water required for proper mixing [11,12]. Furthermore, the concentration of nanocellulose in cementitious composites

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critically influences their performance. Research has shown that low concentrations of nanofibrils can enhance mechanical properties, while higher concentrations may yield detrimental effects [13]. Consequently, nanocellulose often exhibits heterogeneous dispersion within the cement paste when utilized in excessive amounts.The use of nanoparticles as filler materials in concrete, such as nano-clay (NC) [14] and nano-silica (NS) [15,16], has been extensively investigated [17]. These nanoparticles can be classified into two categories: active and inert. Inert nanoparticles enhance concrete properties primarily through nucleation, filler, and bridging effects, while active nanomaterials actively influence the cement hydration process in addition to providing nucleation and filler benefits [18–20]. Microscopic voids within the concrete microstructure can significantly impair engineering properties by introducing gaps and discontinuities in the Interfacial Transition Zone (ITZ). Numerous studies have identified nano-silica as a particularly effective solution to address these challenges [21]. The filling effects provided by nano-silica are fundamental for enhancing the technical properties of concrete [14]. Nano-SiO_2 offers several advantages, including a high specific surface area and superior dispersion, which improve the concrete nanostructure by reducing absorption and enhancing the characteristics of the ITZ. The addition of NS decreases permeability and sorptivity, effectively minimizing capillary pores in the concrete microstructure through the filling of voids with excess hydration products induced by the pozzolanic reactivity of nano-silica [22]. Recent research highlights the benefits of incorporating nano-clay (NC) as a cost-effective additive that enhances concrete strength and durability [23]. NC, consisting of particles approximately 40 nm in size, can serve as a partial substitute for cement, effectively occupying small voids and densifying the porous structure [24]. Studies have demonstrated the impact of nanomaterial substitution on the mechanical properties of concrete; however, notable variability exists regarding the optimal percentage of nanomaterials for replacement [25]. With advancements in nanotechnology, the dual effects of nanoparticles in concrete have led to significant innovations in the construction sector, enhancing the performance characteristics of various concrete elements [26]. Temperature spikes in construction elements can lead to rapid degradation of concrete due to sudden severe stresses, which may develop at rates that compromise structural integrity under impact loads [27]. Thus, addressing the effects of abrupt loading conditions is crucial in the design of concrete structures to mitigate safety risks. Despite the existing body of research, there is a notable lack of studies focusing on the mechanical behavior of concrete that incorporates hybrid nanocellulose materials alongside nano-silica and nano-clay, with limited attention given to their durability and impact resistance. The primary objective of this study is to investigate the properties of hybrid nanocellulose combined with nano-silica and nano-clay in varying ratios within cement mortar mixtures. This research aims to evaluate the mechanical performance and durability of the developed mixtures comprehensively. Additionally, Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) analyses will be conducted to provide deeper insights into the characteristics and performance of the cement matrix incorporating these hybrid nanomaterials. Ultimately, this study seeks to contribute to the development of advanced concrete solutions that offer enhanced mechanical properties and sustainability, thereby addressing pressing challenges in construction materials science.

2. Materials and Methods

In this study, Portland cement was used. The properties of used sand is presented in Table 1. Three different types of nanomaterials were utilized: nanoclay (NC), nanosilica (NS), and nanocellulose. The nanoclay appeared as an off-white powder. The chemical compositions of the ordinary Portland cement (OPC), NC, and NS can be found in Table 3. The nanocellulose was produced by NCTECH®, an Egyptian company, in the laboratory of the National Research Centre. The nanocellulose used in the experiment had 4 -12 nm diameter and a 100 to 450 nm length. Its specific surface area was 80 m^2/gm. The nanocellulose was well-dispersed in a gel, with a 5% concentration. To achieve optimal workability, superplasticizer was added to the proposed mixtures.

Table (1) Properties of Sand:

Particle Size	Fineness Modulus	Specific Gravity
0.5 mm	2.3	2.65 g/cm^3.

Table (2) Properties of NS:

Average particle size	Blaine surface area of
30 nm	45 m^2/g

Table (3) Chemical Composition of used OPC, NS and NC:

Minerals	SiO_2	Al_2O_3	Fe_2O_3	CaO
OPC	20	5.3	3.6	62
NS	99.2	0.14	0.06	0.1
NC	61.24	20.89	1.06	0.16

Mixing procedure and mixture constituents

Table (4) Present the experimental work of this study which containing 12 mixtures. The effect of Nanocellulose was examined over the range up to 1% by weight replacement of cement. The duel effect of NC along with nanocellulose was examined by adding 1.25% NC by weight of cement in some mixes. The mix identifiers "C," "NC," and "NS" represent nanocellulose, nanoclay, and nanosilica, respectively, while the numbers "0," "0.5," "0.75," and "1" denote the nanocellulose replacement ratios.

Table (4): Mix Proportions:

Mix ID	Cement	Sand	%N cell	%N clay	%NS	Water	SP
C0	1	3	0	0	0	0.45	0.02
C0.5			0.5	0	0		
C0.75			0.75	0	0		
C1			1	0	0		
NCC0			0	1.25	0		
NCC0.5			0.5	1.25	0		
NCC0.75			0.75	1.25	0		
NCC1			1	1.25	0		
NSC0			0	0	1.25		
NSC0.5			0.5	0	1.25		
NSC0.75			0.75	0	1.25		
NSC1			1	0	1.25		

Testing method

The compressive strength test was conducted on 50 mm cubes, following the BS EN 12390-3 standard. The flexural strength testing was conducted on prisms 50×50×200 mm according to ASTM C78.

Impact resistance was evaluated using three discs with a diameter of 150 mm and a height of 65 mm. The falling weight test, in accordance with ACI Committee 544, was conducted after 60 days. The test involved repeatedly dropping a 4.5 kg steel ball with from a height of 450 mm under gravity. The ball was released at the center of the upper face of a cylindrical concrete disk (150 mm diameter and 65 mm hight).

Microstructural analysis, SEM and XRD, were also carried out to further characterize the effect of the hybrid nanomaterials.

For water absorption test the samples were casted, and the initial weights of all cubes were recorded. Concrete cubes are submerged in water after 28 days of cure time 24 hours. The initial weight of the concrete cubes is used to compute the quantity of water absorbed.

3. Results and Discussion

Compressive Strength Test

Figure 1 shows the compressive strengths of the mortar with the different nano materials. It was shown that the addition of Nanocellulose to cementitious mortar definitely improved the compressive strength of all specimen. They have a variety of positive effects as the nucleation effect, the filling effect, the bridging effect, and the dispersion effect. The bridging effect occurs when nanocellulose is added, which bridges the nearby hydrated particles as well as the microscopic cracking.

The NC and NS ratios are held constant to assess the major effect of varied quantities of Nanocellulose on concrete compressive strength. The chart above clearly illustrates that for a given NC and NS content, the best percentage of Nanocellulose to achieve maximum compressive strength in concrete is 0.75% by weight of cement. This advantage applies to both concrete days (7 and 28 days). It appears that employing an additional 1% Nanocellulose is not useful. This might be related to the accumulation of nanocellulose particles, which were not evenly distributed throughout the microstructure[28]. However, several investigations have demonstrated that high quantities of nanocellulose produce fibre pockets, which alter mechanical properties [29–31].

From the above figure, it was observed that at a same quantity of NC and NS (1.25%), the use of 0.75% Nanocellulose increased the compressive strength of twenty eight day concrete values by roughly 41% and 81% respectively. The greatest compressive strength were achieved by the mixtures containing 0.75% of Nanocellulose and nano silica (NSC0.75 mix). Hence, it would suggest that the ideal way to get the peak compressive strength of concrete integrating nanocellulose is to include NS. Using Nano-SiO2 particles can increase compressive strength mostly through the filling effect. Additionally, Nano silica is incorporated into the mix to boost the stability of the cellulose nanofibrils, which prevents the fibres from clogging together and hence prevent agglomeration[32]. Also, due to “ their different features, including surface tension, high strength, high specific surface area, nanocellulose have the potential to be used in various cement combinations. Also, hydrophilicity, hyoscopicity, high aspect ratio, and low density have agreat effect.

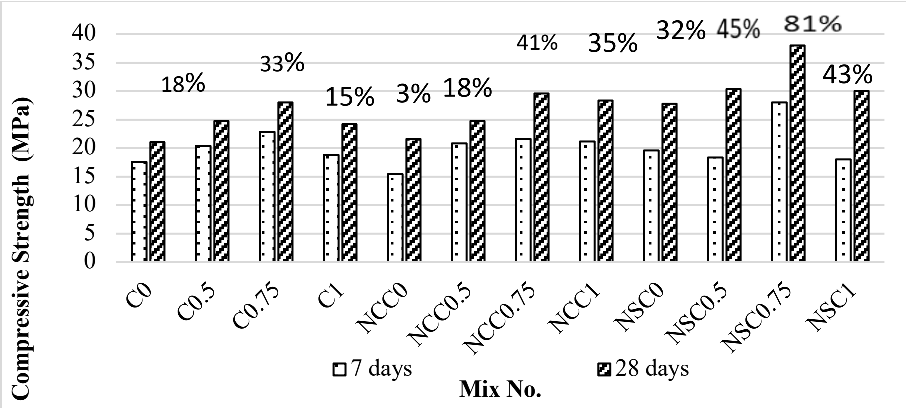


Fig. 1: Compressive Strength Results

Flexural Strength Test

Figure 2 illustrates the flexural strengths of cement mortar containing various nanomaterials at 28 days of age. The results reveal a maximum flexural strength gain of 133% at a replacement ratio of 1.25% nano silica and 0.75% nano cellulose by weight of cement, which is comparable with the compressive strength results. These findings are consistent with prior research, which found that nanocellulose considerably improves the flexural capacity, ductility, toughness, and impact resistance of cement mortar due to its high strength and bonding ability [2,28,29,33].This is a result of the existence of nanofibers, which bridge the first cracks in the matrix and prevent them from propagating. For a constant ratio of 1.25% nano silica, the optimal proportion of Nanocellulose employed to produce optimum flexural strength of cementitious mortar is 0.75% by weight of cement. Furthermore, adding nanocellulose (1%) is ineffective owing to particle aggregation. Furthermore, the cementitious mortar containing hybrid nano cellulose and nano clay increased flexural strength by 100% over the control mix.

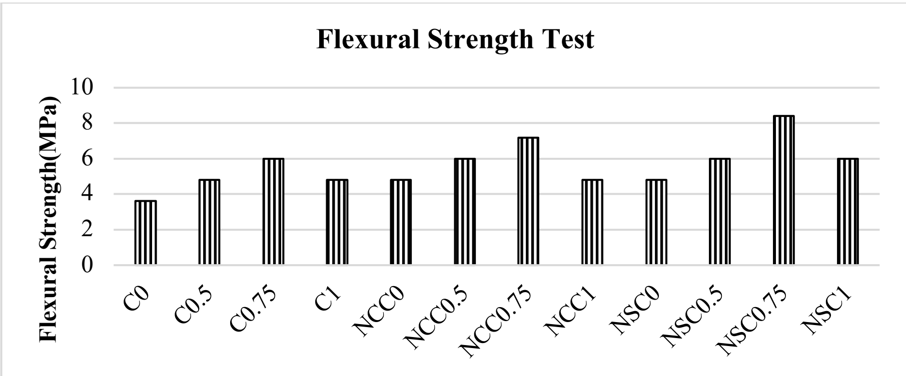


Fig. 2: Flexural Strength Test

It was noticed that, with increasing the replacement ratio of nano cellulose to the mixes incorporating nano clay or nano silica, the strength keeps increasing till reaching ratio of 0.75% nano cellulose. This observation agreed with many previous studies that concluded that the cement degree of hydration increases with the increase in the ratio of nano cellulose. At reaching the replacement ratio to 1% of nano cellulose, the strength obviously decreased. This implies to the agglomeration and theinhomogeneous dispersion of the nano particles that begins to occur. Therefore, it was demonstrated that increasing the flexural strength of cementitious mortar with high nano cellulose ratios requires boosting the dispersion of nano cellulose.

Abrasion Resistance Test

The impact of incorporating nano materials on abrasion resistance is illustrated in Figure 3. The control mix without any nano materials exhibited a weight loss of 4.27 grams due to abrasion. However, when 0.75% nano cellulose was added, the abrasion resistance reduction was 21%. In the case of a hybrid mixture containing 1.25% nano silica and 0.75% nano cellulose, the reduction in abrasion resistance was 14.5%. Similarly, in the mix with 1.25% nano clay and 0.75% nanocellulose, the reduction in abrasion resistance was 45%.

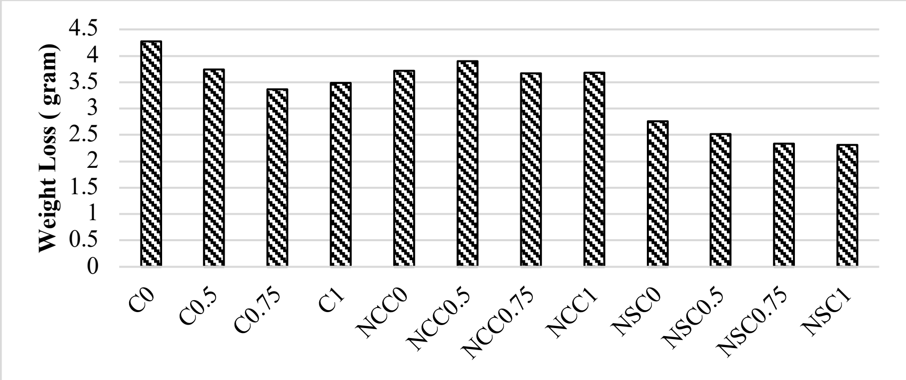


Fig. 3: Abrasion Resistance Test

The findings from Figure 3 indicate that as the percentage of nano cellulose exceeds 0.75%, the weight loss of the concrete sample due to abrasion also increases compared to the control sample. This suggests that a higher percentage of nano cellulose may not be able to adequately fill the voids. Additionally, it was observed that mixes incorporating nano silica exhibited a lower weight loss range compared to mixes with nano clay. Therefore, the utilization of nano silica and nano cellulose had a positive impact on the durability properties. From the results, it can be concluded that the optimal ratio is 1.25% nano silica and 0.75% nano cellulose, as this mixture improved the abrasion resistance compared to the control mix

Impact Resistance Test

The impact resistance of the control mixture and various replacement percentages of nano cellulose, nano silica, and nano cellulose were evaluated in terms of the energy absorbed in the rebound test. As Shown in Figure 4 and Table 5 It was observed that the energy absorbed in the rebound test, required to initiate the initial and final cracks, decreased significantly with an increase in the replacement level of nano cellulose content for all mixtures up to a ratio of 0.75%. However, when the replacement level reached 1%, there was a sharp increase in the energy absorbed in the rebound test, indicating a higher stiffness of the mix, resulting in lower flexibility and reduced absorption of impact energy. The optimal ratio was determined to be 1.25% nano silica and 0.75% nano cellulose, as the energy absorbed in the rebound test decreased due to the decreased stiffness of the nano particles, leading to improved plasticity of the mortar and absorption of a significant amount of impact energy

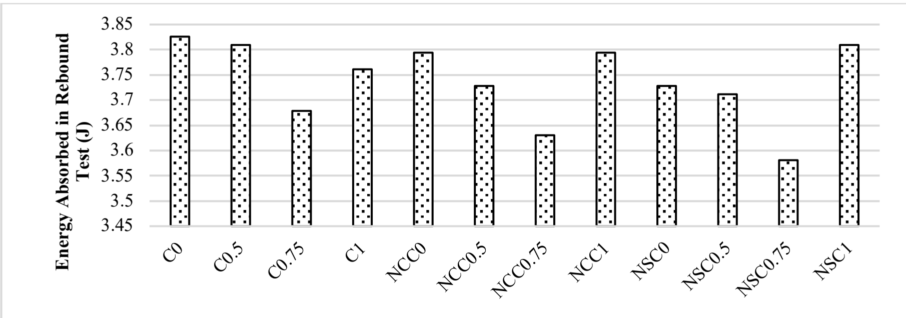


Fig. 4: Impact resistance Test

Table (5): Impact resistance results under drop weight test

Mix no.	C0	C0.5	C0.75	C1	NCC0	NCC0.5	NCC0.75	NCC1	NSC0	NSC0.5	NSC0.75	NSC1
Average	12	12	15	13	13	14	16	13	14	14	17	12
Standard Deviations	0.00	0.47	1.41	0.94	0.47	2.16	1.41	0.47	2.16	1.89	0.47	0.47
Coefficient of Variation	0.00	3.82	9.43	7.07	3.72	15.43	8.84	3.72	15.43	13.16	2.77	3.82

Absorption Test

The results of the water absorption test are depicted in Figure 5. The findings demonstrate that the water absorption values for specimens incorporating nano silica at various concentrations of nano cellulose were lower compared to those containing nano clay mixtures. This can be attributed to the high reactivity of the nano silica particles, which react with calcium hydroxide to generate C-S-H gel at an early stage, enhancing pozzolanic activity and improving the microstructure. The increased concentration of hydration products promotes concrete compaction, resulting in a denser and more homogeneous microstructure. Consequently, the nano silica particles effectively filled the spaces between the cement particles. On the other hand, as the replacement level of nano cellulose content increased for all mixtures, the water absorption values decreased significantly until the ratio reached 0.75%. However, when the replacement level reached 1%, there was a notable increase in water absorption values, indicating a less refined pore structure. This could be attributed to the dispersion of the micro particles and the significant absorption of free water by 1% nanocellulose, which reduced the availability of water for the hydration process, resulting in microscopic cracks and voids[34, 35].

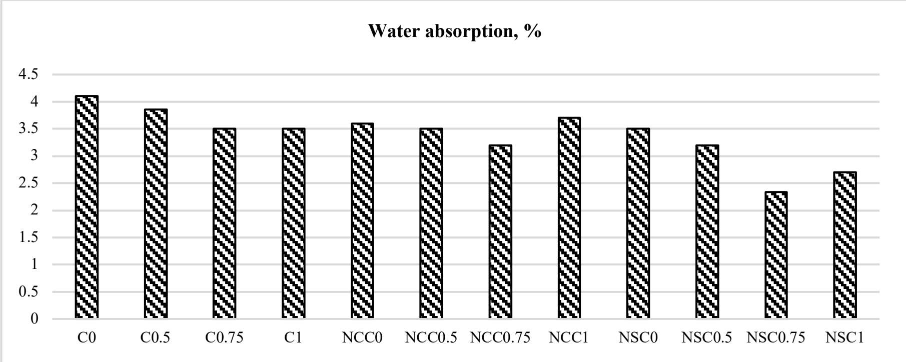
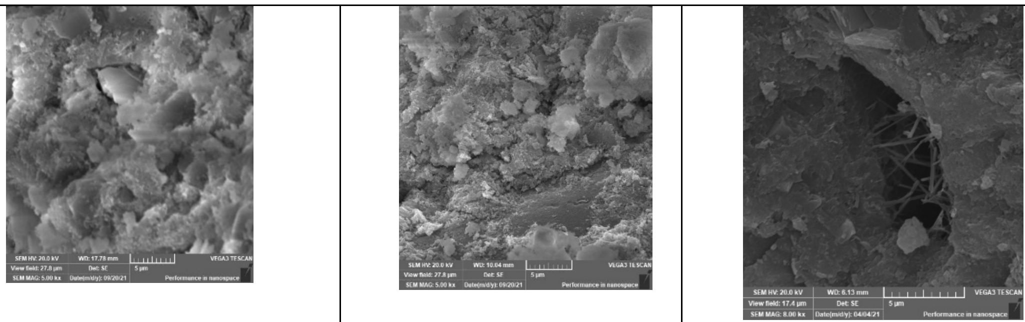


Fig. 5: Absorption Test for all mixes at 28 days

It was demonstrated that when the replacement level of nano cellulose content increased for all mixes, the water absorption values decreased significantly until the ratio reached 0.75%. When the replacement level of nano cellulose content reached 1%, there was a dramatic increase in water absorption values, indicating that the pore structure was not well refined. This might be related to the dispersion of the micro particles. Furthermore, at a concentration of 1% nanocellulose, it absorbs a significant amount of free water, reducing the amount of water involved in the creation of $Ca(OH)2$ via the hydration process, resulting in microscopic cracks and voids.

Scanning Electron Microscope

To better analyse the attained outcomes and evaluate the effect of blending nano cellulose with nano clay and nano silica, SEM test were executed. In figure 6(a), the sem analysis of the control mix was shown. The non-homogenous impact of the packed particles with permeable microstructure was seen resulting in excess pores. In figure 6(b), the sem analysis of the NCC0.75 mix was shown. The presence of nano clay together with nano cellulose in the cement matrix improved the uniformity of mixes, leading to a more packed matrix. Even though agglomerations of the nano clay is detected in the mix NCC0.75, the microstructure was remained tight and packed. Filling effect causes void ratio to be reduced and hence increasing the matrix's density. Additionally, the interaction of nano clay particles together with nano cellulose with the remaining CH from the cement hydration process, leads to a greater amount of CSH gels. In figure 6(c), the sem analysis of the NCS0.75 mix was shown. SEM study in figure 6(c) shows that the bridging effect has the ability to inhibit the formation of tiny cracks in microstructure and to improve both the strength and durability performance. As compared to many other aspects that affect the strength of concrete by nanocellulose, the bridging effect has the biggest influence on boosting its mechanical properties.



(a) Mix C0	**(b)Mix NCC0.75**	**(c)Mix NCS0.75**

Fig. 6: SEM at 28 days for mixes (a) C0, (b) NCC0.75, (c) NCS0.75

X-ray diffraction (XRD) analysis:

XRD shows the crystallographic structure and phase composition of the investigated cement based materials. In the context of nanotechnology-enabled innovations in cement, XRD can provide valuable insights into the effects of nano cellulose, nano silica, and nano clay additives on the performance and durability of cement based composites.

From figure 7, and 8, representing the control mix (without nano addition) as compared to the mix of the greatest compressive strength containing 0.75% of Nano cellulose and nano silica (NSC0.75 mix), and by comparing the XRD patterns of the mixes it is clearly to identify the changes in the crystalline phases. The XRD patterns helped in identifying the presence of extra reaction products, such as C-S-H gel, which were not clearly present in the pure cement. The degree of pozzolanic reaction can be assessed by monitoring the disappearance of the (Ca (OH)2) peaks associated with unreacted cement phases (in general, the characteristic diffraction peaks for calcium hydroxide (Ca(OH)2) in XRD analysis were observed at approximately 18.3° and 34.5° 2θ. These peaks correspond to the (001) and (002) crystallographic planes, respectively) and the appearance of increase in the C-S-H peaks corresponding to hydrated products. It worth noting that the calcium silicate hydrate (C-S-H) is a more complex phase with a variable composition and amorphous structure, making its identification through XRD challenging. The amorphous nature of C-S-H means that it does not exhibit well-defined diffraction peaks. However, some studies have reported broad humps or shoulders around 29° to 32° 2θ, which can be connected with the presence of C-S-H in cementitious materials.

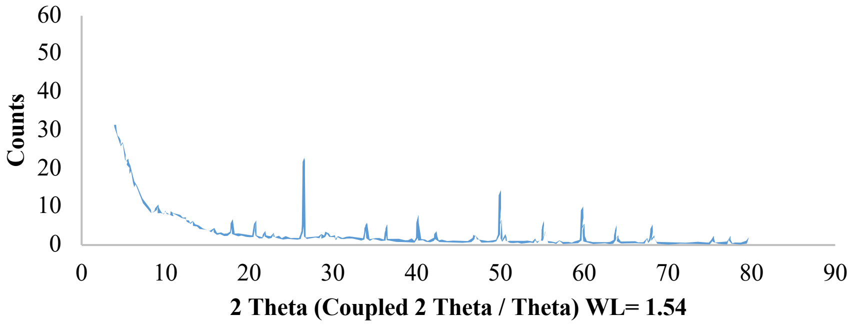


Fig. 7: XRD for mixes C0

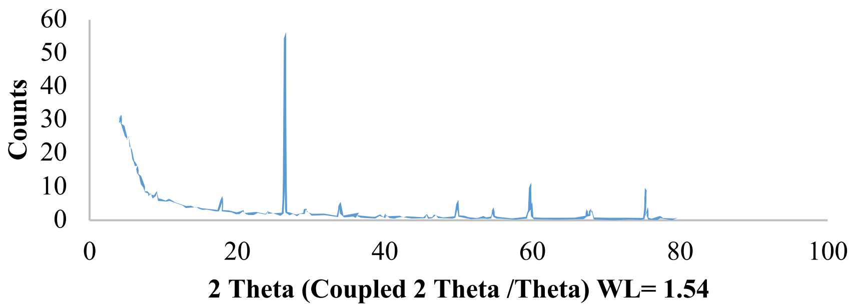


Fig. 8: XRD for mix NCC0.75

4. Conclusions

In conclusion, the incorporation of nanocellulose and nano silica into cement mortar has demonstrated a significant positive impact on the compressive and flexural strengths of concrete. Optimal proportions of 0.75% nanocellulose and 1.25% nano silica, by weight of cement, have been identified as effective for achieving maximum compressive and flexural strengths. It is crucial to highlight that excessive concentrations of nanocellulose can lead to agglomeration and non-uniform dispersion of nanoparticles, which may negate performance benefits. Furthermore, the synergistic combination of nanocellulose with nano clay has shown enhancements in flexural strength. Additionally, the inclusion of these nanoparticles has improved abrasion resistance, impact resistance, and water absorption properties of concrete, with the optimal ratio for maximum abrasion resistance being 1.25% nano silica and 0.75% nanocellulose by weight of cement. The presence of nano clay and nanocellulose within the cement matrix has also contributed to improved mix uniformity and mitigated the formation of microcracks, thereby enhancing overall strength and durability. These findings underscore the potential of nanoparticles to significantly augment the engineering properties of various cement compositions. Future research efforts should focus on optimizing nanoparticle dispersion to unlock even greater performance enhancements. Overall, this study substantiates the transformative potential of nanoparticles to drive the cement industry toward producing more sustainable and high-performance concrete solutions.

Conflicts of interest

The authors certify that they have NO affiliations with or involvement in any organization or entity with any financial interest, or non-financial interest in the subject matter or materials discussed in this manuscript.

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