INTERNATIONAL JOURNAL OF SUSTAINABLE CONSTRUCTION ENGINEERING AND TECHNOLOGY
ISSN: 2180-3242 e-ISSN: 2600-7959

IJSCET

Vol. 16 No. 1 (2025) 76-91
https://publisher.uthm.edu.my/ojs/index.php/ijscet

Enhanced Mechanical Properties of Mortar Reinforced with Recycled and Depolymerized Nylon Microfibers

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DOI: https://doi.org/10.30880/ijscet.2025.16.01.007

Article Info

Received: 14 October 2024
Accepted: 13 June 2025
Available online: 30 June 2025

Keywords

Ɛ-caprolactam, nylon 6, polymeric mortars, depolymerization, mechanical properties

Abstract

This study explores the potential of recycled nylon 6 to improve the mechanical properties of mortar modified through chemical additives and the reinforcement of nylon microfibers, referred to as the chemical and physical recycling methods, respectively. The chemical recycling approach involved the depolymerization of nylon into ε-caprolactam monomers, while the physical method utilized recycled nylon microfibers derived from waste. The investigation emphasized evaluating the reinforcement potential of pure depolymerized ε-caprolactam, without other additives, in mortars. Laboratory-produced mortars incorporated these additives, and their flexural, tensile, and compressive properties were meticulously examined. Additives, including the chemical additive ε-caprolactam and nylon microfibers, were incorporated at concentrations of 0.10%, 0.15%, 0.25%, and 0.50% relative to the cement weight. The purity of the depolymerized monomer was verified using Fourier transform infrared spectroscopy analysis. Mechanical tests of the modified mortars (with chemical additive or microfibers) were conducted at 7 and 28 days of curing, to assess the impact of the additives incorporation on the mechanical properties of polymeric mortars. The additive concentration significantly influenced the mortar performance. The optimal compressive strength was achieved with a 0.25% concentration of both chemical additive and microfibers, while tensile strength improved markedly with 0.25% of ε-caprolactam. Similarly, flexural and tensile strengths were maximized with a 0.10% concentration of nylon microfibers. Notably, compressive strength for mortars with 0.25% ε-caprolactam derived from depolymerized nylon waste was comparable to those with commercially sourced monomers. These findings highlight the viability of recycling nylon waste as a sustainable strategy for producing effective additives in polymeric-modified mortars. This approach emphasizes the potential of reusing low-value plastic waste for innovative applications in the civil construction industry.

1. Introduction

Polymer fibers have been used in construction materials since the 1950s. It encompasses three main applications: (a) polymer concrete, (b) polymer-impregnated concrete, and (c) polymer-modified concrete [1]. In this context, nylon fibers are synthetic, elastic, and robust thermoplastic polymers. Nylon 6 (polycaprolactam, NF-6) is widely used across various industrial sectors, including toothbrushes, hosiery, fishing lines, and gear components [2]. NF-6 is intrinsically heat-stable, hydrophilic, relatively inert, and resistant to natural decomposition. Classified as a high-purity semicrystalline thermoplastic, nylon exhibits a highly organized molecular structure, providing industrial-grade resistance to pH fluctuations and various thermal conditions. Additionally, it exhibits high rigidity and solvent resistance. Nylon 6 fibers stand out for their superior properties compared to other synthetic and natural fibers. These properties include high toughness, substantial elongation capacity, and exceptional abrasion resistance. Its structural stability derives from hydrogen bonds [3]. However, previous studies [4,5] have indicated its susceptibility to hydrolytic degradation and the potential for reuse through depolymerization to the basic monomer Ɛ-Caprolactam. The feasibility of this process depends on the technology (equipment, catalysts, and operating conditions) and the associated costs [6,7].

Most studies have focused on the physical recycling of nylon, specifically on reducing the size of the fiber for direct incorporation into the mix, evaluating various ratios. Ɛ-caprolactam was applied as an oligomer along with an additional additive to improve multiple properties in the concrete simultaneously [8]. The addition of nylon fibers has been shown to effectively increase tensile and flexural strength, depending on the percentage and size of the fiber added [9]. Nurazuwa et al. [10] have suggested that optimal compressive strength is achieved by adding 1% fibers of 0.65 mm in diameter and 15 mm in length. Under these conditions, they achieved a compressive strength of 54.93 MPa at 28 days of curing. Good compaction was ensured to avoid excessive air accumulation and, consequently, reduce porosity in the concrete.

However, studies such as those by Srimahachota et al. [11] have demonstrated a decrease in compressive strength with an increase in the percentage of added nylon; similar behavior was observed by Kumaresan et al. [12], who reported a reduction in compressive strength by increasing the dose of nylon fibers from 0.5% to 3%. While incorporating 0.5% and 1% recycled nylon fibers significantly reduced compressive strength, the addition of 0.5% fibers resulted in a 20.2% increase in tensile strength. On the other hand, the addition of 1% nylon fibers negatively affected the permeability of the concrete [13]. Ahmad et al. [14] identified an optimal blend with 50% recycled aggregates, 0.5% nylon fibers, and 20% silica fume, as the nylon fibers significantly reduced crack dispersion, improved both compressive and tensile strength, and increased acid attack resistance by 8%.

Most research to date has focused on enhancing the mechanical properties of polymer concrete by adding fibers as physical additives [15]. However, comparing this approach to the chemical recycling of nylon 6, where the polymer is broken down into its monomer form and introduced into concrete in a more elemental state, offers a potentially transformative influence on the material's quality and performance. This method also presents a circular recycling option for materials at the end of their life cycle, specifically those containing nylon 6, a polymer notoriously challenging to process and often considered unrecyclable. This study aims to propose a viable reuse strategy for nylon 6 in the construction industry, which continuously seeks improvements in strength and durability. By pursuing chemical recycling, we explore new avenues for improving concrete properties and contribute to waste reduction and the promotion of a circular economy. This research highlights the potential benefits of transforming waste nylon 6 into valuable additives, offering a more sustainable approach to the material used in construction.

Therefore, here we evaluate the feasibility of using recycled nylon 6 to improve the properties of the concrete. In addition, it compares the impact of chemical recycling, achieved through depolymerization with Ɛ-Caprolactam, and physical recycling, which involves recycled microfibers. Various concentrations of recycled nylon-6 (0.10%, 0.15%, 0.25%, and 0.50% by cement weight) were examined to determine their influence on the compression, bending, tensile, and permeability properties of concrete. The mechanical properties of the reinforced cement were evaluated after 7 and 28 days of curing to analyze changes in the concrete's surface structure. This research aims to provide insights into the comparative effects of chemical and physical recycling of nylon 6 on concrete performance. It focuses on their potential applications in concrete technology.

Nomenclature	
NF-6	Nylon-6 Fiber
NM	Nylon Microfiber
CA	Chemical Additive
Fs, Ts, and Cs	Flexural, Tensile, and Compressive strengths.

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2. Materials and Methods

Table 1 presents the chemical requirements of Type I Portland cement used in this study, comparing the manufacturer's specifications with limits defined by ASTM C150. The values include magnesium oxide (MgO), sulfur trioxide (SO_3), loss on ignition (PF), and insoluble residue (IRI).

Table 1 *Chemical composition of Portland cement Type I compared to ASTM C150 specifications*

Chemical requirements %	Portland cement type I	ASTM C 150
Magnesium oxide (MgO)	2.0 - 4.0	6.00 maximum
Sulfur trioxide (SO_3)	1.8 – 2.5	3.00 maximum
Loss on ignition, (LOI)	0.1 – 2.5	3.00 maximum
Insoluble residue (IRI)	< 1.5	1.50 maximum

Table 2 *Physical properties of Portland cement Type I compared to ASTM C150 standards*

Physical requirements	Portland cement type I	ASTM C 150
Specific gravity (g/cm^3)	3.10 – 3.15	-
Fineness (cm^2/g)	3000 – 3700	2600
Autoclave expansion (%)	0.0 – 0.2	0.80 maximum
Initial Vicat setting time (min)	140 – 190	45 – 375

Nylon 6 fibers (NF-6): The nylon 6 microfibers used in this study were obtained from the wear of recycled fishing lines. Before use, these materials underwent a disinfection process, followed by the selection of nylon 6-containing parts. The selected components were then transformed into microfibers through cutting and grating. The resulting microfibers, with lengths below 5 mm and an average diameter of approximately 0.035 mm, were characterized using appropriate analytical techniques.

Aggregates: For the fine component, the study used white natural silica sand as the fine aggregate, characterized by a maximum moisture content of 2.00%, a bulk density of 1500 kg/m^3, and a maximum particle size of 0.5 mm. The consistent size and shape of the particles contribute to a homogeneous mix, facilitating the development of a concrete structure that is both uniform and robust.

2.1 Ɛ-Caprolactam Production by Nylon 6 Depolymerization

This study focused on two types of samples: fishing lines (Sample A), derived from waste produced by the fishing industry, and recycled fishing gear components (Sample B), derived from industrial waste. Sample A comprised nylon filaments frequently used in commercial fishing operations, while sample B included plastic components from recycled fishing gear and industrial equipment. Both samples underwent a preparatory conditioning process before depolymerization, following the methodology proposed by Elgegren et al. [16]. This process used potassium carbonate as a catalytic agent to accelerate the depolymerization of polymer-based materials. These samples were chosen for their availability and representativeness of polymeric materials prevalent in industrial and consumer waste.

In the experimental setup, each 10 g sample was combined with 30 g of potassium carbonate (K_2CO_3) inside a 1-liter stainless steel reactor, maintaining a ratio of 1:3, as illustrated in Figure 1. The reactor was equipped with two outputs: one connected to a water-cooling condenser supplied by SEDAPAR, S.A., and the other to a CPS vacuum pump (nominal power: 1/2 HP). A calibrated vacuum gauge monitored pressure levels between –0.80 and –0.92 bar.

The experiments were carried out in temperatures ranging from 13°C to 250°C, regulated by an AUTONICS digital temperature controller (model TCN4L-24R) and a calibrated Type "K" thermocouple from TESPRO. The reactor was placed inside a quartz sand bath throughout the experiments to minimize rapid temperature fluctuations. The depolymerization process took place for approximately 2 hours. After each experiment, the product was cooled to ambient temperature and pressure. It was then carefully removed from the reactor with a spatula and transferred to a petri dish for further analysis.

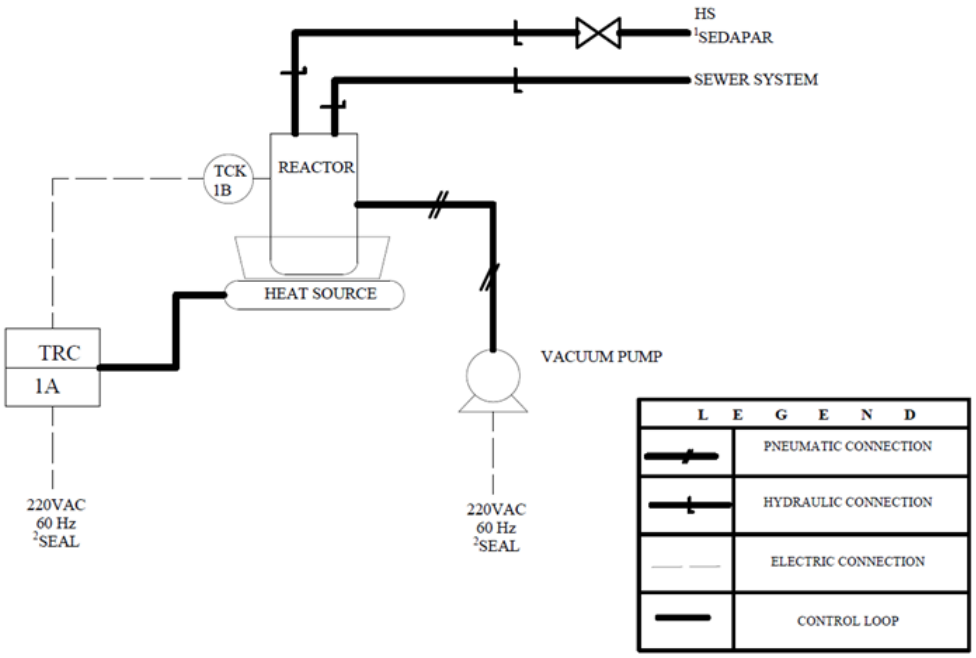

Fig. 1 *Depolymerization reactor to convert NF-6 to Ɛ-caprolactam (the characteristic monomer). [1]SEDAPAR. Drinking water network; [2]SEAL. Electric power network (Arequipa, Perú)*

2.2 Characterization of the Obtained Monomer (Ɛ-Caprolactam)

The resulting solid product was analyzed using Fourier transform infrared spectroscopy (FTIR) with a Perkin Elmer Frontier FTIR/NIR spectrophotometer. The scanning range was set between 4000 cm^{-1} and 650 cm^{-1}. An applied force 80 was used, and a Universal ATR accessory type, UATR Diamond/ZnSe crystal. The setup included UATR with 3 jumps, and correlation was applied during the test. The identification process used the Polymer Start spectral database, following the procedures suggested by Villar [17]. The results were then compared with commercial Ɛ-Caprolactam monomer acquired from Lab Solution International SRL.

2.3 Formulation of Mixtures for the Analysis of Concrete Properties

This study evaluated the mechanical properties of concrete after incorporating recycled and conditioned Nylon 6 (NF-6) and ε-Caprolactam, derived from the depolymerization process. The methodology adhered to ASTM C496/C496M, C293/C293M, and C109/C109M standards to ensure consistency and reliability in evaluating the mechanical properties. These standards ensured methodological rigor in the evaluation of the mechanical characteristics of the prepared concrete samples.

The concrete formulations included YURA® cement, standardized silica sand, water, and recycled NF-6 polymer. The NF-6 was subjected to physical (denoted as NM) and chemical (denoted as CA) modifications. These treated polymers were used as additives in the creation of polymer-reinforced mortars. The precise ratios and mixtures of these materials were documented following ASTM standards. Table 3 details the experimental configurations used to examine each mechanical property of interest.

Table 3 *Mix formulations for evaluating the mechanical properties of concretes incorporating recycled Nylon 6 microfibers and Ɛ-Caprolactam obtained by depolymerization*

Mechanical property assessed	Materials used for mortar	Chemical treatment	Monomer mass (CA) added, g	Percentage of addition, %	Physical treatment	Mass of recycled polymer (NM) added, g	Percentage of addition, %
Flexural (Fs)	Cement: 800g Standardized sand: 2400g Water: 125g	CA0.00F	0	0	NM0.00F	0	0
		CA0.10F	3.3283	0.10	NM 0.10F	3.3283	0.10
		CA0.15F	4.9950	0.15	NM 0.15F	4.9950	0.15
		CA0.25F	8.3333	0.25	NM 0.25F	8.3333	0.25
		CA0.50F	16.7085	0.50	NM 0.50F	16.7085	0.50

Traction (Ts)	Cement: 3000 g Standardized sand: 9000 g Water: 1700 g	CA0.00T	0	0	NM 0.00T	0	0
		CA0.10T	7.0070	0.10	NM 0.10T	7.0070	0.10
		CA0.15T	10.5158	0.15	NM 0.15T	10.5158	0.15
		CA0.25T	17.5439	0.25	NM 0.25T	17.5439	0.25
		CA0.50T	35.1759	0.50	NM 0.50T	35.1759	0.50
Compressive (Cs)	Cement: 247g Standardized sand: 679g Water: 125g	CA0.00C	0	0	NM 0.00C	0	0
		CA0.10C	1.0521	0.10	NM 0.10C	1.0521	0.10
		CA0.15C	1.5789	0.15	NM 0.15C	1.5789	0.15
		CA0.25C	2.6341	0.25	NM 0.25C	2.6341	0.25
		CA0.50C	5.2814	0.50	NM 0.50C	5.2814	0.50

Testing standards: Based on ASTM C293/C293M, ASTM C496/C496M, and ASTM C109/C109M.

2.4 Flexural-Tensile-Compressive Strength Tests

Experimental prototypes were manufactured to evaluate bending, compression, and tensile strength, incorporating CA and NM at concentrations of 0.10%, 0.15%, 0.25%, and 0.50%, calculated based on the cement weight.

(a) Flexural Strength was determined in accordance with ASTM C293 [18] standards using 40 × 40 × 160 mm beams. The beans were prepared with a water-to-cement ratio (W/C) of 0.50 and standardized sand in a 3:1 ratio relative to the cement weight. Tests were carried out using an INSTRON universal testing machine with a capacity of 100 kN.

(b) Tensile strength was evaluated following ASTM C496 [19] standards using concrete cylinders (100 mm diameter and 200 mm height). The cylinders were prepared with a W/C ratio of 0.50 and standardized sand in a 2.75:1 ratio regarding the cement weight. For the preparation, CA was dissolved in water, while NM was graded to particles smaller than 1000 micrometers (micro sprayed). The tests were carried out using an ELE INTERNATIONAL universal testing machine with a capacity of 2224 kN.

(c) Compressive Strength was assessed according to ASTM C109 [20] standards using 50 × 50 × 50 mm concrete cubes. These cubes were produced with a W/C ratio of 0.50 and standardized sand at a 2.75:1 ratio regarding the cement weight. Compressive strength tests were conducted on an INSTRON universal testing machine with a capacity of 100 kN.

2.5 Sample Morphology

Surface morphology was analyzed as a critical property to assess the impact of CA and NM additives. For this reason, the concrete morphology was examined with the addition of CA and NM. The samples, analyzed after 28 days of healing, included CA0.00 (0%), CA0.10 and NM0.10 (0.10%), as well as CA0.25 and NM0.25 (0.25%). Scanning electron microscopy (SEM) was used to obtain images at various magnifications and scales. This technique enables detailed analysis and characterization in both 2D and 3D at micro and nanoscale levels. The Scios 2 LoVac FIB-SEM scanning electron microscope was used for this purpose.

2.6 Setting Time

Cement setting time, the period required for a mixture of cement paste and water to go from a liquid to a solid state is critical. It is imperative that the initial adjustment occurs gradually and that unnecessary delays in the final adjustment are avoided. These times are essential for assessing the normal progression of hydration reactions within the cement paste. Optimal setting times enhance cement performance and improve construction efficiency. Gypsum, included in cement, plays a crucial role in controlling setting time; however, factors such as cement fineness, water/cement ratio (W/C), and additives used in different concentrations of CA and NM (0%, 0.10%, 0.15%, 0.25%, and 0.50%) also influence these durations.

Therefore, the Vicat Needle was used to determine the initial and final setting times of the cement paste. The results, including time (in minutes) and needle penetration (in millimeters), are detailed in Tables 4 and 5. According to ASTM C150 [21], the initial setting time for Portland Type I cement paste without additives ranges from 45 to 60 minutes, while the final setting time is approximately 10 hours.

Table 4 *Results of penetration measurements for setting time tests with different percentages of CA and NM using the Vicat needle*

Time (min.)	Measurement in Vicat (mm)								
	CP	CA				NM			
		0.1	0.15	0.25	0.5	0.1	0.15	0.25	0.5
0	2	2	1.5	2	1	1	1.5	1	1
30	1.5	2.1	1.5	2	1	1.3	2	1	1
45	1.5	1.9	2	2.3	2.5	1.5	3	2	1.9
60	1.7	3	2.5	2	2	1.5	4.2	2	2.1
75	2.9	5	2	1	1.9	2.5	5.5	2	2.5
90	3.9	2.5	2	3.2	2	3.1	6.5	3	3
105	4.5	5	2	4	2.5	5.5	6.1	4	5
120	16.5	4	5.1	5	2.8	17.5	21	8	8.5
135	26.5	21	16.5	9	4.5	25	33	15	25
150	32.5	26.5	23	24.3	3.9	37.5	36.5	27.8	32
165	32	32.5	28	32.5	6	37	34.3	35	34.8
180	37.9	35	34.6	34	11.5	38	36	38.5	36
195	38.5	36.5	36.2	37	23	38	31.9	38	36
210	38.1	34.5	35.5	38.5	30	37.5	36	37	35.5
225	39	36.2	36.5	38.5	34.5	38.9	36	38	37
240	38.5	37.5	37		36	37.5	37	36.5	36.2
255	39.5		36.9		37.3	38.5	36.5	37.5	36.5
270	39.1		36.9		38.5	38.9			
285					38				

**CP = Control Cement Paste*
**CA = Chemical Additive*
**NM = Nylon Microfiber*

2.7 Statistical Analysis

The compression, bending, and tensile strength tests were performed in triplicate. Data derived from the use of chemical additives (CA) and nylon microfiber (NM) in various proportions were analyzed to determine their mechanical properties (compressive, flexural, and tensile strength). Bartlett and Kolmogorov tests were used to assess the normality and homogeneity of variance, which are crucial for the analysis of variance. Next, a unidirectional analysis of variance (ANOVA) was performed to investigate the impact of these additives on mechanical strengths (Fs, Ts, and Cs for flexural, tensile, and compressive strengths, respectively).

The post hoc pairwise comparison using the Games-Howell method assessed differences between mean values of all these experimental data, considering uneven variances. The significance level was set at $p < 0.05$ for this study. The dataset was analyzed using Origin Pro 2023 (Origin Lab) software. The mean error calculations obtained using the Games-Howell method were illustrated as error bars in Figs. 3, 4, 5 and 6.

3. Result and Discussion

Fig. 2 presents the characterization spectra of samples derived from depolymerization together with a commercially acquired Ɛ-Caprolactam sample. This figure illustrates the similarity of the signals obtained at various wavelengths between commercial Ɛ-Caprolactam and the laboratory-depolymerized samples. Specifically, sample A exhibits a similarity of 97.1%, while sample B shows 96.5% similarity with the database in the FTIR equipment software. This result indicates a remarkable purity of 99% of the monomers obtained compared to the commercial monomer. The parallelisms observed in the spectra confirm the efficiency of the depolymerization process in producing high-purity monomers.

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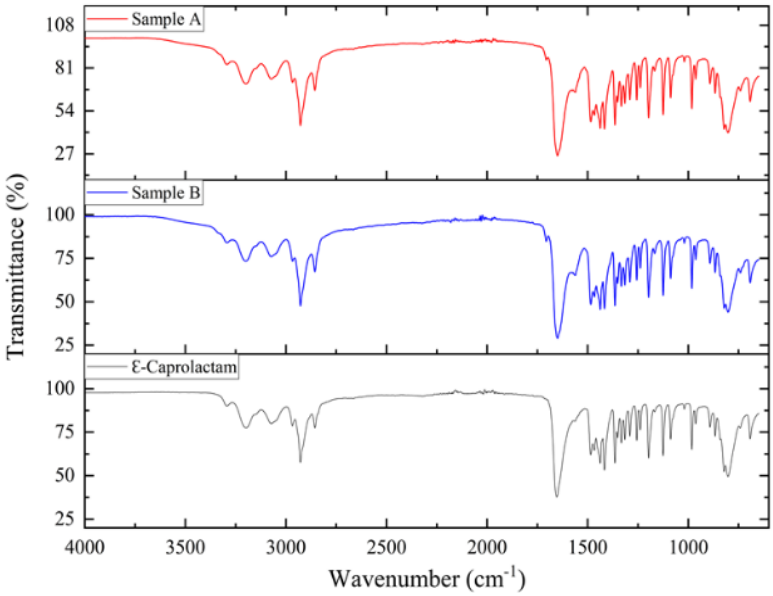

Fig. 2 *The characterization spectrograms and commercial spectrograms of Ɛ-Caprolactam (black curve) are compared with those obtained from recycled samples of NF-6: Sample A (fishing lines) and sample B (recycled fishing gear materials)*

3.1 Flexural Strength (Fs)

Evaluating the flexural strength of concrete elements is crucial for ensuring structural safety, regulatory compliance in design, and extending the structure's service life. It also prevents potential failures, reduces costs, and maintains long-term structural integrity. Flexural strength is a critical mechanical property for many structures, including beams, columns, slabs, plates, prefabricated elements, and bridge structures [22]. In general, the evaluation of bending is crucial in any concrete structure where there is the possibility of bending moments inducing loads, which requires the guarantee of safety and structural integrity [23].

The control samples with nylon microfibers (NM0.00F) showed flexural strengths of 6.3349 MPa at 7 days and 8.0414 MPa at 28 days. There is a trend of increasing flexural strength at 28 days with NM0.10F (0.10%), reaching a value of 8.5899 MPa, followed by a decrease with the concentration of NM0.15F (0.15%), obtaining values of 7.7673 MPa. Subsequently, for both concentrations of NM0.25F (0.25%) and NM0.50F (0.50%), increases of 0.1849 MPa and 0.2038 MPa, respectively, are observed. Even at the highest concentration of 0.50%, the flexural strength values at 7 and 28 days were lower than those achieved with 0.10% NM, which showed a 6.84% increase over the control sample after 28 days of curing.

As for the chemical additive, it is observed that the flexural strength shows a decreasing trend compared to the CA0.00F control sample for the samples cured at 28 days. Figure 3 shows reductions in flexural strengths of 0.4179 MPa and 0.0188 MPa with CA0.10F (0.10%) and CA0.15F (0.15%), respectively. The trend changes with an increase of 0.0815 MPa in the concentration of CA0.25F (0.25%), after which a decrease of 0.3109 MPa is observed again in the concentration of CA0.50F (0.50%). Note that in the case of using the chemical additive for 28 days of curing, the trend in most concentrations is decreasing, except CA0.25F (0.25%) with a value of 7.6862 MPa, which is still 8.21% below the CA0.00F control sample at 28 days of curing.

Flexural tests were conducted at 7 and 28 days since no plasticizing additives were used to accelerate the curing process. It is considered that after 28 days, sufficient and stable strength is achieved for concrete, according to ASTM C293. The results of the flexural tests conducted using the center-point method are presented. The flexural strength modulus at failure was evaluated for both the chemical additive and the nylon microfibers, as shown in Figure 3. The results suggest that, at most concentrations, nylon microfibers are more effective than the chemical additive in enhancing flexural strength. In this regard, Oszar et al. [24] indicate that flexural strength is higher with the use of nylon microfibers compared to other fibers. Using 1% and 0.5% nylon microfibers by volume, they achieved flexural strengths of 3.75 MPa and 4.34 MPa, respectively, at 28 days. A difference of 15.73% is observed between the two values; However, both values are lower than those obtained in our case study (8.5899 MPa) when 0.1% of the nylon microfibers was used.

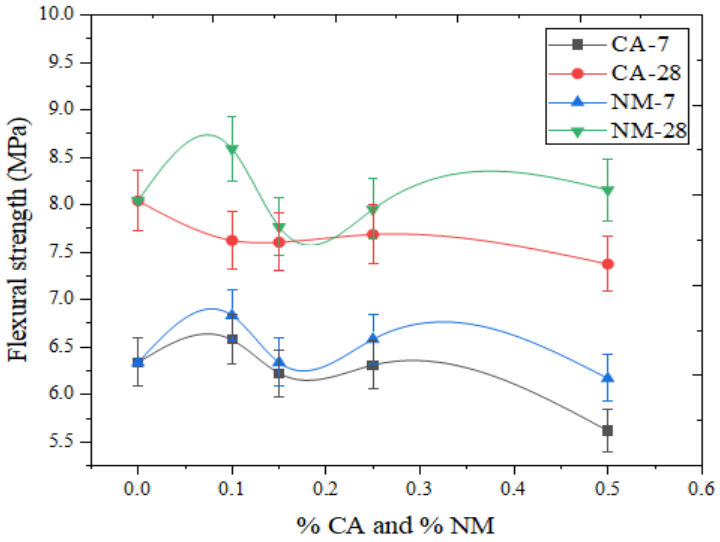

Fig. 3 *The impact of varying concentrations of the chemical additive and nylon microfibers on the flexural strength property*

The optimal flexural strength values were 6.8326 MPa and 8.5899 MPa at 7 and 28 days, respectively, obtained using a 0.10% NM concentration. Therefore, adding 0.10% increases flexural strength by 7.9% and 6.8% during 7 and 28 days of curing, respectively. Thus, based on the evaluated references, it can be inferred that the decrease in the curve after reaching the optimal point can be attributed to the depletion of the concrete properties due to an overload or stress in the polymer matrix of the concrete [25].

Similarly, Munadrah et al. [26] determined a maximum increase of 24.2% in flexural strength, with a strength value of approximately 5.5 MPa working with 0.75% nylon microfibers compared to their control sample. In our study, the maximum increase in flexural strength was 6.84% compared to the control sample, NM0.10F (0.10%). Our beam had a flexural strength of 8.59 MPa, which is higher than those mentioned in previous reports [24, 26]. On the other hand, Dang et al. [27] conducted a study that revealed that using a hybrid fiber blend with nylon produced flexural strength results of approximately 8.20 MPa. This result closely matches the findings for NM0.10F (0.10%), with a flexural strength of 8.59 MPa. In particular, Dang et al. [27] used hybrid fibers composed of 0.5% nylon and 1% amorphous metal fibers, while the present investigation exclusively incorporated recycled nylon-6.

3.2 Tensile Strength (Ts)

The tensile, or diametric compression test, was conducted to assess the tensile strength of the concrete samples with the additive. The tensile strength was determined through diametrical compression tests according to ASTM C496 [20], applying load to the cross-sectional area of each specimen.

Tensile and compressive strengths are typically correlated. However, Qureshi et al. [28] observed an inverse relationship, noting that the presence of nylon fibers tends to reduce compressive strength. Fig. 4 illustrates a significant increase in tensile strength with the addition of 0.10% nylon microfibers. This finding aligns with studies in the literature, such as the review by Jawad et al. [15] on concrete admixtures, and the work by Bhat et al. [29], who compared the tensile strength of concrete reinforced with steel fibers, polymeric concrete (silicone rubber), and polymeric concrete with steel fibers after 28 days of curing. They observed improvements in tensile strength of 38.85%, 21.65%, and 47.72%, respectively, highlighting the effectiveness of fibers reinforcement.

The tensile strength modulus was evaluated for concrete samples containing nylon microfibers and a chemical additive. The results indicate that samples with nylon microfibers generally outperformed those with the chemical additive. In the control samples without additives (NM0.00T), the tensile strength increased from 1.337 MPa at 7 days to 1.826 MPa after 28 days of curing. For samples with 0.10% nylon microfibers (NM0.10T), the tensile strength reached a peak of 2.496 MPa. A slight decrease was observed at 0.15% concentration (NM0.15T), where the tensile strength dropped to 2.017 MPa When the highest concentration of 0.50% was used, the tensile strength achieved (2.418 MPa) was slightly lower than the peak value at 0.10%. These findings suggest that lower concentrations of nylon microfibers (10%) provide an optimal balance between performance and material efficiency for practical applications.

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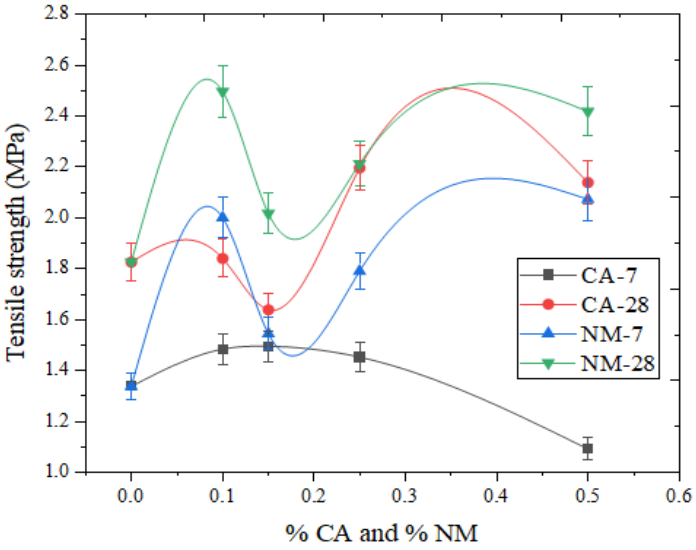

Fig. 4 *Effect of varying concentrations of chemical additive and nylon microfibers on the tensile strength of concrete*

Regarding the use of the chemical additive, a different trend in tensile strength is observed compared to tests with the physical additive. Unlike CA0.00T, which starts at 1.826 MPa in 28 days, there is an increase of 0.014 MPa with the CA0.10T (0.10%) concentration. The trend changes with a decrease of 0.202 MPa at the CA0.15T (0.15%) concentration, followed by an increase of 0.558 MPa in CA0.25T (0.25%), and then another drop of 0.057 MPa in CA0.50T (0.50%). It is noteworthy that, for CA samples, most concentrations exhibit lower tensile strength than the concrete with the nylon microfibers, except for CA0.25T (0.25%), which achieved 2.196 MPa, similar to values observed for mortars with nylon microfibers at the same concentration. Even at the highest percentage of CA0.50T (0.50%), the tensile strength of 2.139 MPa remained lower than that achieved at 0.25% (CA0.25T).

The optimal tensile strength values of 2,000 MPa and 2,496 MPa at 7 and 28 days were achieved with a concentration of 0.10% of the nylon microfibers. This concentration resulted in a remarkable 50% and 37% increase in tensile strength during the respective 7 and 28 days of curing. In this context, our findings align with the observations of Sejal et al. [30] that incorporating recycled fibers and materials improves mechanical strength by limiting crack propagation. This restriction minimizes the entry of water and other substances into the mortar. These authors used discarded carpet fibers, including nylon and polypropylene textile fibers, and compared the results with cement containing virgin nylon fibers. They found a bridging mechanism in the cracks and a reduction in pore size, resulting in improvements in tensile strength.

3.3 Compressive Strength (Cs)

Fig. 5 illustrates the results of the compression tests conducted in accordance with ASTM C109 standards. Compressive strength moduli were evaluated for concrete incorporating a chemical additive and nylon microfibers. The results indicate that concrete with nylon microfibers generally exhibits higher compressive strength compared to concrete with chemical additives, as detailed in the subsequent discussion. Samples without nylon microfibers (NM0.00C) exhibit compressive strength values of 9.654 MPa and 14.553 MPa at 7 and 28 days of curing. At NM0.10C (0.10%), a slight decrease of 0.026 MPa was observed. This was followed by increases of 1.531 MPa and 7.350 MPa at NM0.15C (0.15%) and NM0.25C (0.25%), respectively. However, a significant drop of 8.841 MPa was recorded at the NM0.50C (0.50%) concentration.

On the other hand, the compressive strength of concrete with chemical additive shows a different trend compared to those with nylon microfibers. The sample without additives, CA0.00C, shows a compressive strength value of 14.553 MPa, then shows a growth trend for the concentrations CA0.10C (0.10%), CA0.15C (0.15%) and CA0.25C (0.25%), with values of 0.531 MPa, 0.618 MPa, and 4.533 MPa, respectively. Finally, it experiences a drop of 4.002 MPa for the concentration of CA0.50C (0.50%). In particular, even with the highest percentage of additive (0.50%), the compressive strength value is 16.2314 MPa, which is lower than the value achieved with 0.25% of the chemical additive, which measures 20.234 MPa. Unlike the findings for flexural and tensile strengths, where 0.10% was optimal, the highest compressive strengths were achieved with 0.25% for both chemical additive and nylon microfibers. This disparity indicates that mortar reaches its maximum load-bearing capacity at this percentage. Before or after this point, the strength values experienced a decrease. This decrease suggests that the admixture reaches its stress threshold at maximum load, either before or after the concrete initiates deformation.

It should be noted that the time at which the admixture reaches its stress threshold, in relation to the onset of mortar deformation, varies not only with the type of load (bending, tensile, or compression) but also depends on the specific structural configuration or intended application of the concrete.

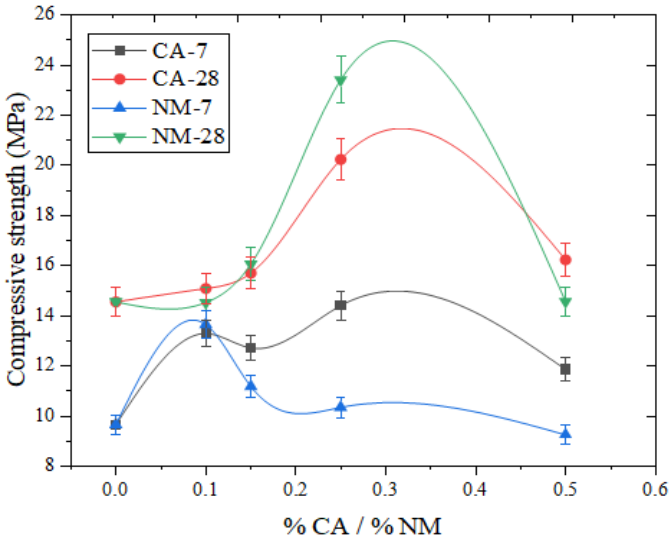

Fig. 5 *The effect of increasing the percentage with chemical additive and Nylon microfibers on compressive strength properties*

The optimal compressive strength values are 10.343 MPa and 23.408 MPa at 7 and 28 days of curing, respectively, obtained using a 0.25% nylon microfibers concentration. Thus, it is shown that this concentration leads to a 7% and 61% increase in compressive strength during 7 and 28 days of curing, respectively. This growth is observable in the compressive strength curve at an NM concentration of 0.25%, showing a decrease after reaching the optimal point. Interestingly, concrete containing a CA and cured at 7 days exhibited higher compressive strength values than those containing nylon microfibers for additive values of 0.15% to 0.5%. This behavior had not been previously observed for flexural and tensile strengths. Therefore, we can infer that incorporating the chemical additive improves compressive strength during the early curing stages.

Ali et al. [13] studied recycled nylon fibers (25–40 mm) in concrete with nylon 6 concentrations from 0.1% to 1.0%. After 28 days of curing, they recommend 0.1% and 0.25% of these fibers, as they observed 7% and 2% increases in compressive strength, respectively. This behavior aligns with the study by Farooq et al. [31], who indicate that fibers contribute to compressive strength and warn that increasing the amount of fibers (from 0.25%) will decrease compressive strength. They noted a 5% increase in crack resistance, reaching approximately 33 MPa at 0.25% concentration. Similarly, Kumaresan et al. [12] demonstrated that compressive strength decreases as fibers concentration increases. They used recycled fishing net fibers with concentrations of 0.5% to 3% and recycled carpet fibers ranging from 0.25% to 3%, all with lengths ranging from 12 to 30 mm.

Amran et al. [32] investigated the impact of various types of fibers, including nylon fibers with lengths ranging from 10 to 60 mm, on foamed concrete in compressive strength tests. Their results revealed a marked improvement in compressive strength, reaching maximum values up to 2.5 times higher than those of foamed concrete without fibers. On the other hand, Nurazuwa et al. [10] investigated different types of fibers incorporated in self-compacting concrete, ranging from 15 to 20 mm in length. They observed an increase in compressive strength at 28 days of curing, particularly when 0.50% monofilament nylon fibers were used, achieving a value of 40.97 MPa. This property represented a substantial 68.74% increase in the maximum compressive strength value compared to the control.

Abbas et al. [33] reported that the incorporation of 12 mm nylon fibers along with alumina powder in concentrations ranging from 10% to 50% for both admixtures in concrete increased compressive strength with the increasing concentration of nylon fibers (from 0% to 2%). Specifically, using the minimum concentration of alumina dust led to an increase within the range of about 41 MPa to 43 MPa. However, the compressive strength results did not show a clear and consistent trend, attributed to the filling effect of alumina powder within the pores of the reinforced concrete.

Additionally, Ahmad et al. [14] indicate that the use of 20 mm nylon fibers as reinforcement in recycled concrete aggregates results in an approximate increase in compressive strength (both in terms of percentage increase and MPa increase) for samples at 7 days of curing, ranging from 21.5 MPa to 24 MPa. However, over 28 days of curing, along with an additional 20% volume of silica fume, the compressive strength increased to about

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34-38 MPa. This suggests the possibility of further improving the increase in compressive strength by simultaneously using additives with nylon microfibers.

While the compressive strength values reported by Ahmad et al. [14] exceed those obtained in our study using 5 mm fibers (23.41 MPa), differences in fiber dimensions and concentrations between the two studies may explain this variation. It is also noteworthy that the percentage increases reported by Ahmad et al. [14], ranging from approximately 2.5% to 11.6%, are lower than those obtained in our study, which registered approximately 60.85% and 39.04% for NM 0.25% and CA 0.25%, respectively, at 28 days after curing.

Statistical analysis of all concretes (Fig. 3–5) showed that flexural strength means and variances were similar for concretes with nylon additives, regardless of chemical or physical treatment. In contrast, the averages and variations of tensile and compressive strengths in all concrete samples (including chemically treated admixtures and nylon microfibers) demonstrated significant differences in varying proportions at 7 and 28 days of curing.

For samples that incorporated CA after 7 days of curing, significant distinctions emerged in mean values of flexural, tensile, and compressive strength in all additive percentages relative to samples without additives. After 28 days of healing, a contrasting trend manifested. Specifically, non-significant differences were observed in the mean flexural and compressive strength values, while significant differences persisted in the mean tensile strength values.

For the NM-containing samples, insignificant differences were observed in mean flexural strength values at 7 and 28 days. However, notable distinctions were identified in the mean values of tensile and compressive strengths. This statistical analysis emphasizes the discernible effects of chemical and physical treatments and duration of curing on the mechanical properties of mortar reinforced with nylon, CA, and NM. The thorough examination of the flexural, tensile, and compressive strengths at the different stages of curing provides valuable information on the material's behavior, contributing to a deeper understanding of the performance of these modified concretes.

In addition, tests were carried out with depolymerized CA and commercial CA at concentrations of 0%, 0.10%, 0.25%, and 0.50% at 7 and 28 days of curing. Figure 6 illustrates the results, intending to analyze the similarity between the two types of CA and their impact on the compressive strength of concrete.

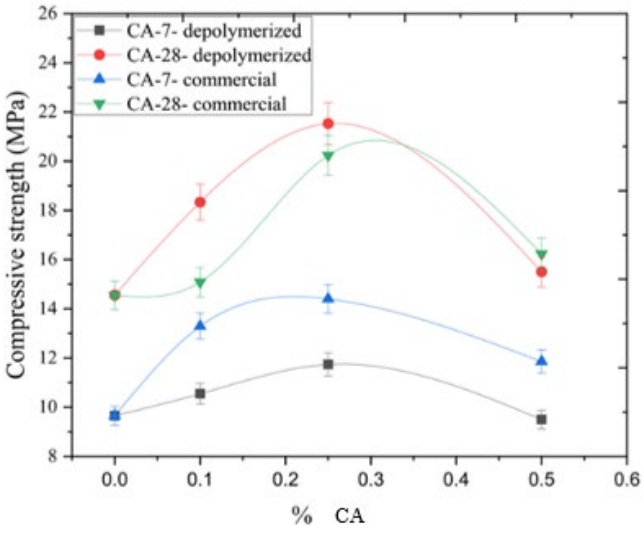


Fig. 6 *Comparison of compressive strength with increasing percentages of chemical additives derived from nylon 6 depolymerization and commercially available chemical additive*

At concentrations of 0.10% and 0.25% CA, whether depolymerized or commercial, a noticeable increase in compressive strength is observed at 28 days of curing. Specifically, there is an improvement of 3.78103 MPa and 6.97569 MPa for the depolymerized CA and 0.0257 MPa and 5.1503 MPa for the commercial CA, respectively. The highest compressive strength values were reached at 0.25% of the AC, reaching 20.2338 MPa for the commercial CA and 21.52849 MPa for the depolymerized CA, the latter exhibiting superior results.

Fig. 6 demonstrates that the lab-synthesized additive outperforms the commercially obtained chemical additive in compressive strength. These findings strongly suggest that monomers derived from depolymerized nylon waste are effective concrete admixtures with significant environmental benefits.

3.4 Evaluation of the Adjustment Time

Table 5 *Results of the initial and final setting times of cement with various concentrations of CA and NM using the Vicat needle*

Sample	Percentage (%)	Sample Code	Initial setting time (min)	Final setting time (min)
Cement paste	0	CP	135	270
Nylon-6 monomer (CA)	0.1	CA0.1	150	225
	0.15	CA0.15	165	240
	0.25	CA0.25	165	225
	0.5	CA0.50	210	285
Nylon-6 microfibers (NM)	0.1	NM0.1	135	210
	0.15	NM0.15	135	255
	0.25	NM0.25	150	255
	0.5	NM0.50	135	255

Table 5 shows the variations in initial and final setting times of cement paste with different percentages of nylon monomer and microfibers. The control cement paste (CP), without additives, exhibited an initial setting time of 135 minutes and a final setting time of 270 minutes. With the addition of nylon monomer (CA) at a concentration of 0.1%, the initial setting time was extended to 150 minutes, while the final setting time was reduced to 225 minutes. As the concentration increased to 0.15% and 0.25%, the initial setting time stabilized at approximately 165 minutes. Although the final setting time fluctuated, it remained at 225 minutes for the 0.25% concentration. At a concentration of 0.5%, the initial setting time increases significantly to 210 minutes, and the final setting time is extended to 285 minutes.

For nylon-6 microfibers (NM), unlike monomers, their addition does not significantly alter the initial setting time, which remains within the range of 135 to 150 minutes. However, the final setting time increases substantially with microfiber concentrations above 0.15%, reaching 255 minutes at 0.15%, 0.25%, and 0.5%. This indicates that while the initial hydration process of cement paste remains unaffected by microfibers, their presence considerably prolongs the final hardening phase.

The variation in cement setting times with increasing concentrations of nylon monomer (ε-caprolactam) suggests a complex interaction mechanism between cement components and additives. At lower concentrations, the monomer likely catalyzes hydration processes, accelerating the initial setting phase. This behavior may result from its interaction with the cement's primary hydration products, such as tricalcium silicate (C_3S) and tricalcium aluminate (C_3A), which are responsible for the early development of strength. At higher monomer concentrations, the monomer interferes with the formation of the cementitious matrix's microstructure, inhibiting the catalytic effect observed at lower concentrations. This leads to a delay in the final setting time, which is made possible by the formation of a passivating layer on the surface of the cement particles. This layer could reduce water availability, inhibiting hydration reactions during the later stages of the process, thus slowing down the final setting.

Conversely, the addition of nylon 6 microfibers predominantly influences the final setting time, possibly by forming a physical network within the cementitious matrix. This network of fibers modifies the microstructure by decreasing permeability and restricting water diffusion, which delays the cement's complete hardening. Unlike the monomer, microfibers exert a primarily physical influence, prolonging the final setting stage without significantly affecting the initial setting time.

Future research should investigate rheological properties and time-dependent behavior (e.g., plasticity, flow, and dimensional stability) to gain a deeper understanding of the influence of nylon 6 additions on the microstructure and cement setting process. This would provide a more complete understanding of how these additives affect the performance of the final cement product.

3.5 pH Assessment

Carbonation in concrete can significantly affect its strength and durability, increasing its susceptibility to wear and corrosion. Evaluating pH levels is essential for understanding the potential carbonation properties of the material. Samples containing CA and NM were analyzed for their reaction to phenolphthalein, by observing changes in the mortars. The samples exhibited a pronounced magenta-pink hue (figures not shown), indicating

the presence of carbonation. This observed coloration usually signifies the presence of carbonation within a pH range of 12 to 13.

Consequently, our findings suggest that the concrete possesses favorable properties for corrosion resistance. These results align with the established understanding that pH levels significantly influence material properties. Maintaining an appropriate pH is crucial to ensure concrete durability and strength, thereby optimizing its performance over its service life. Our study shows that an alkaline pH range between 12 and 13 enhances steel reinforcement protection against corrosion, as detailed in Table 6. Furthermore, our research underscores the importance of maintaining optimal pH levels. This lays the foundation for future studies on carbonation properties to further improve concrete's corrosion resistance. Additionally, our study recorded an initial pH value of 13.05 for mortar incorporating nylon microfibers and a chemical additive, which remained stable throughout the curing process.

Nylon microfibers and chemical additives caused a slight reduction in alkalinity during curing, with an average decrease of 0.3 in both cases between 7 to 28 days. In addition, at a concentration of 0.25%, pH values remained consistent at 13.10 and 12.85 for both NM and CA at 7 and 28 days, respectively. These values indicate that the studied admixtures do not compromise the pH stability of the concrete samples.

Table 6 *pH levels during curing time with nylon microfibers and chemical additive*

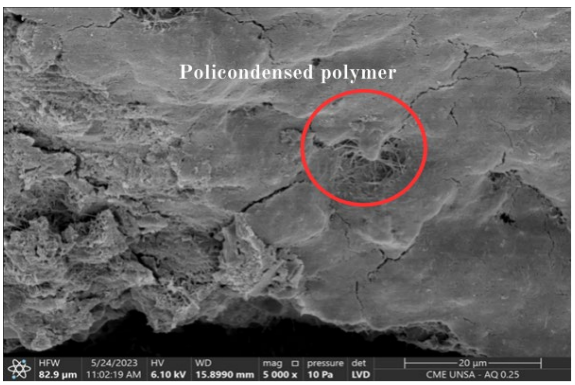
Percentage of addition, %	CA7	NM7	CA28	NM28
0	13.05	13.05	13	13
0.1	12.9	13.2	12.7	12.85
0.15	13.25	13.2	12.85	12.9
0.25	13.1	13.1	12.85	12.85
0.5	13.15	13.2	12.75	12.9

3.6 Sample Morphology

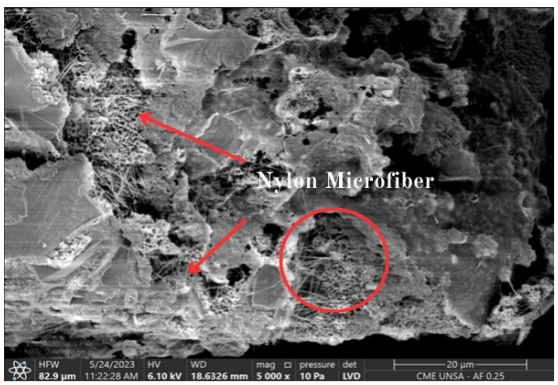
The morphology of concrete samples cured for 28 days was examined using scanning electron microscopy (SEM). The micrographs reveal well-defined polycondensed polymer structures and interwoven nylon fibers. These features are crucial for understanding the distribution of nylon microfibers and the chemical additive (monomer Ɛ-caprolactam) within the concrete matrix. This dispersion was prominent in samples with enhanced compressive, flexural, and tensile strength. Figure 7 compares concrete surfaces containing 0.25% chemical additive (Fig. 7A and 7C) and 0.25% nylon-6 microfibers (Fig. 7B and 7D).

At 5000× magnification (Figures 7A and 7B), nylon fibers were clearly visible in the NM0.25 sample. At the same time, they were absent in the NM0.10 sample (image not shown) due to the lower fiber concentration in that sample. In addition, the CA0.25 sample, containing the nylon monomer Ɛ-caprolactam, exhibited unexpected regions with interlocking fiber structures. At 40000× magnification (Figures 7C and 7D), cross-linked fiber structures were observed in both the CA0.25 sample (0.25% nylon monomer) and the NM0.25 sample (0.25% nylon microfibers). These fibers played a crucial role in improving the material's durability by forming bridge-like structures, thus preventing the concrete from deteriorating with shorter cure times, as noted by Liang et al. [34].

The mechanical strength improvements observed in Figures 3-5 are attributed to the effective distribution of fibers within the polymer concrete matrix. The greater fibers content in the NM0.25 sample (Figure 7B) compared to the CA0.25C sample (Figure 7A) explains the superior compressive strength of CA0.25C, emphasizing the reinforcing role of nylon microfibers in the mortar structure.



(a)



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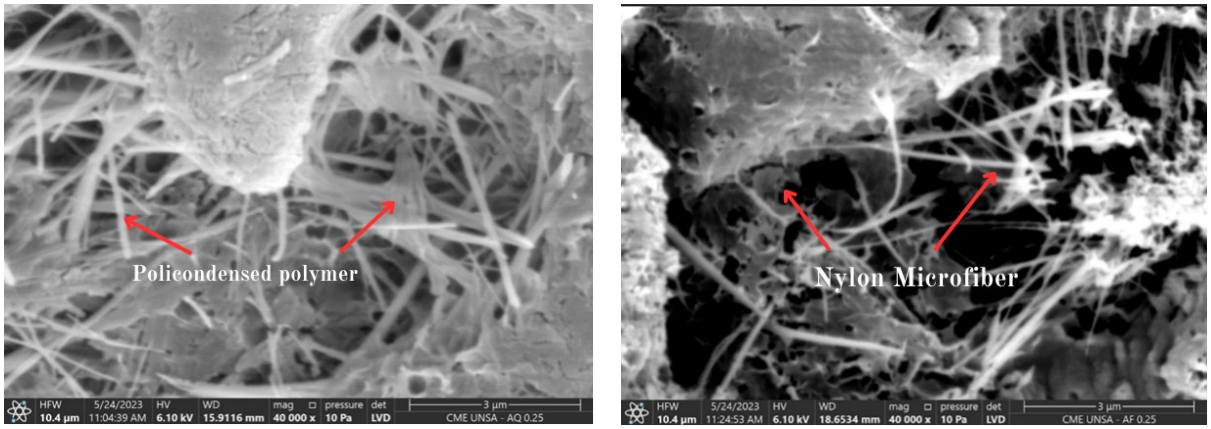

Fig. 7 *SEM micrographs of samples with chemical additive (CA0.25) (a and c) and nylon microfibers (NM0.25) (b and d). Images a and b were captured at 5000× magnification, while images c and d were taken at 40000× magnification*

The formation of microfibers in samples with dissolved Ɛ-Caprolactam (Figure 7C) as an additive in concrete preparation, can be attributed to its polymerization, catalyzed by the presence of water. This process involves the hydrolysis of Ɛ-Caprolactam into aminocaproic acid, followed by its linear polycondensation, as reported in previous studies [35, 36]. In this sense, the improved tensile strength and compressive strength results of samples containing the chemical additive (Fig. 4 and 5) are attributed to fibers formed during the concrete mixing and setting. Polyakov et al. [8] also reported that this monomer acts as a water-soluble wetting agent on the surface of cement particles and aggregates.

4. Conclusion

This study investigated the effect of recycled and depolymerized nylon microfibers at various concentrations as additives on the mechanical properties of reinforced mortars. Nylon-6 was successfully depolymerized into its monomer, Ɛ-caprolactam, using a custom-designed stainless-steel reactor, achieving a monomer purity of 99%, comparable to commercial standards.

Compression tests performed on concrete containing recycled and depolymerized nylon revealed that adding 0.25% recycled nylon-6 microfibers resulted in higher compressive strength (23.41 MPa) compared to samples with an equivalent concentration of the chemical additive (20.23 MPa). Incorporating nylon microfibers and the chemical additive increased the compressive strength of the concrete by 60.85% and 39.04%, respectively, after 28 days of curing compared to concrete without additives. This improvement is attributed to the microfiber network that reinforces the concrete matrix. Therefore, this study demonstrates that recycled nylon-6 microfibers and the monomer from chemical recycling can effectively improve mortar properties.

Furthermore, the percentage of chemical additives and nylon-6 microfibers significantly influences the mechanical properties of polymeric mortar. Using 0.25% microfibers or chemical additives yields optimal compressive strength in mortar. The addition of 0.10% nylon 6 microfibers enhances flexural and tensile strength. Furthermore, the addition of 0.25% chemical additive improves tensile properties.

Furthermore, experiments with 0.25% Ɛ-Caprolactam from the depolymerization of nylon 6 and commercial sources showed similar compressive strength values. The concrete with depolymerized additive achieved a maximum compressive strength of 21.53 MPa, slightly higher than the one with the compared commercial additive (20.23 MPa). These results highlight the potential of depolymerized nylon 6 waste as an effective additive in polymer concrete. Our findings confirm the feasibility of repurposing plastic waste into valuable additives for the civil construction industry, promoting a more sustainable and environmentally friendly process. Future studies should analyze the modulus of elasticity in concrete reinforced with depolymerized and recycled nylon fibers to better understand their mechanical benefits.

Acknowledgement

The authors thank the National University of San Agustin de Arequipa with GRANT CONTRACT N° TP IB-60-2021-UNSA for the financial support of this work. GRSB also thanks the National Council for Technological and Scientific Development of Brazil-CNPq (grant 305287/2022-2) for its scholarship.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

All authors: conceptualization, methodology, software, validation, formal analysis, research, writing (original draft, writing, reviewing, and editing), data curation resources, and fundraising. Giancarlo R. Salazar-Banda: also, supervision and project management.

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