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Recycled cement mortars reinforced with PVDF nanofibers synthesized by electrospinning

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ABSTRACT

Due to the significant environmental problems caused by obtaining raw materials for construction, it has become very important to replace natural fine aggregates with recycled fine aggregates (RFAs) in the production of cementitious materials. However, the use of RFAs decreases the mechanical properties of cementitious materials. The addition of nanofibers (NFs) is a promising way to counteract this disadvantage. In our study, polyvinylidene fluoride (PVDF) NFs were synthesized by electrospinning and added to mortar mixtures in proportions of 0.025, 0.05, and 0.1 % by weight of cement in order to evaluate their effect on the microstructure, flowability, compressive strength, flexural strength, total porosity, and resistance to chloride-ion (Cl^-) penetration of those mortars. The results showed that the PVDF NFs had diameters of 788 nm and they physically interacted with the hydration products. In terms of compressive and flexural strength, the addition of 0.1 % NFs caused an increase of 12 and 23 % respectively, and a decrease in porosity of 3 % in relation to recycled mortar. In addition, the recycled mortars with NFs saw a 20 % increase in chloride-ion permeability, which could help to broaden the applications of recycled mortar. The PVDF NFs in recycled mortars counteract the negative effects of using RFAs, in addition to improving cementitious materials.

1. Introduction

Portland cement mortar is widely used in the construction industry for floor leveling, slope stabilization, and especially in the construction of masonry, such as for adhering partitions, plastering, and decorative work [1,2]. One of the most significant environmental problems caused by the construction industry is the excessive extraction of natural resources by dynamiting hills or collecting aggregates from rivers, which seriously damage ecosystems [3]. In addition, this industry generates large amounts of construction and demolition waste (CDW) worldwide, and much of this waste is disposed of in illegal landfills, causing soil contamination and again seriously damaging ecosystems. It has been estimated that approximately 10,000 tons of CDW are generated annually worldwide [4]. The U.S. produced about 569 million tons in 2017 [5], and in the European Union, approximately 835 million tons are produced each

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year [6]. Unfortunately, only a few countries have recycling standards that apply to their reuse. Due to these problems, it has become a matter of great importance for the scientific and engineering community to replace natural aggregates (NAs) with recycled aggregates (RAs) in cementitious materials in order to achieve a more environmentally friendly construction industry [7]. In the production of mortars, it has been found that the use of recycled fine aggregates (RFAs) causes a decrease in certain properties, such as compression strength, bending, porosity, and durability. This is because these materials have already fulfilled one life cycle, which makes them less dense, more porous, and more fragile, and means they might contain undesirable particles (contaminants) [8,9]. Therefore, before using them to produce recycled mortars, it is important to first evaluate their mechanical and durability properties. One of the most important tests for durability is resistance to chloride ions (Cl^-) because cementitious materials are largely associated with reinforcing steel, and when the chloride concentration in the mortar exceeds a certain threshold value, depassivation of the reinforcing steel occurs and it starts to corrode, causing structural damage and economic losses [10,11]. To increase the mechanical and durability properties of the mortar, and to counteract the negative effect of the use of RFAs, some fixes have been used, such as reducing the water/cement ratio with the help of superplasticizers, adding supplementary cementitious materials (silica fume, fly ash, slag), introducing chemical additives and incorporating fibers [12–14]. It has been reported in the literature that fibers can function as reinforcement in cementitious materials, providing resistance to internal stresses, reducing the generation of cracks by delaying the fracture of the element, and reducing porosity and shrinkage, among other things [15]. Indeed, fiber-reinforced cementitious materials have been found to increase the mechanical properties and durability of cementitious materials [16].

In most investigations, the diameter of the fibers ranges from 200 μm to ~0.4 cm, with very few investigations having assessed fibers with diameters on the nanometer scale [17]. The application of nanotechnology in the construction industry is relatively recent and its application could help to improve the mechanical and durability properties of cementitious materials. Unfortunately, it has not been exploited due to the high costs involved in synthesizing nanomaterials and the difficulty of manipulating nanomaterials. Despite these disadvantages, nanomaterials provide better properties than materials at the macroscopic scale, so it is important to develop new research into their potential applications [18]. Therefore, the challenge for researchers is to produce nanomaterials with economical, ecological, and simple methods that can produce nanomaterials on a large scale.

Among the useful nanomaterials, polymeric nanofibers (NFs) have attracted much attention from the scientific community due to their advantages. These have, for example, a high surface/volume ratio, good viscoelastic properties, are light, highly porous, and have good mechanical properties [19]. Nguyen and associates in 2020 synthesized Nylon 66 NFs using an electrospinning technique and added them, in various percentages, to cement pastes. These additions resulted in an increase in the flexural, compressive, and toughness characteristics of 30, 8, and 49 %, respectively [20]. Chinchillas and associates in 2020 synthesized recycled polyethylene terephthalate and polyacrylonitrile (PAN) NFs using the electrospinning technique and incorporated these into cement mortars. This resulted in an increase in compressive strength of 26 %, an increase in flexural strength of 86 %, and a decrease in drying shrinkage of 93 % [21]. Several methods are used for the synthesis of polymeric NFs, such as blowspinning, spinning centrifugation, and electrospinning [22–25]. Among these methods, electrospinning is a technique that is easy to develop and use, and it is possible to vary the parameters to obtain NFs with different properties. In this technique, an electrical charge is applied to a flow of polymeric solution, which causes viscoelastic stretching of the solution and the deposition of NFs on a collecting plate [26,27]. Some of the polymers used in the synthesis of NFs include polyacrylic acid [28], polyvinyl pyrrolidone [29], polyvinyl alcohol [30], PAN [31], polypropylene [32], and polyvinylidene fluoride (PVDF) [33]. Among the wide variety of polymers used to synthesize NFs, PVDF has attracted a lot of attention from researchers because it is a highly inert thermoplastic polymer with high mechanical properties and chemical resistance, and good thermal stability, it is easy to process, and has a high piezoelectric coefficient [34,35]. Synthesizing PVDF NFs is thus a good option for reinforcing and improving the properties of cementitious materials.

In this work, PVDF NFs were synthesized by electrospinning and added to recycled mortar mixtures in proportions of 0.025, 0.5, and 0.1 % by weight of cement. The effect of the NFs on the microstructure, mechanical properties (compressive and flexural strength), flowability, total porosity, and resistance to chloride permeability of the mortars was evaluated.

2. Materials and methods

2.1. Materials

The materials necessary for the synthesis of nanofibers were the following: PVDF with a molecular weight of 534,000 g/mol, dimethylformamide (DMF) at 99.85 % purity and acetone at 99.99 % purity. All three reagents were purchased from Sigma Aldrich. The RFA (obtained after mechanical recycling using a jaw crusher at the Autonomous University of Sinaloa [UAS–Mexico]), Cemex® brand Portland cement type III-30R, and distilled water were used to make mortars. The aggregate grain size and properties of the materials are provided in Fig. 1 and Table 1.

2.2. RFA

To obtain the RFA, cylindrical specimens of concrete waste were collected from a construction laboratory; these had a compressive strength of 24 MPa. The cylinders were thoroughly cleaned with water and then crushed for 20 min in a Maneklal global exports jaw crusher (Model JS-0804) to obtain 1 cm RFA particle sizes. This material was then subjected to a second crushing process for 30 min using a falcon ball crusher (Model 7518081 PA56C), incorporating 0.8–1.2 cm steel balls. After this, the RFA particles were approximately 0.075 cm in size. Finally, the RFA was washed twice to remove contaminants.

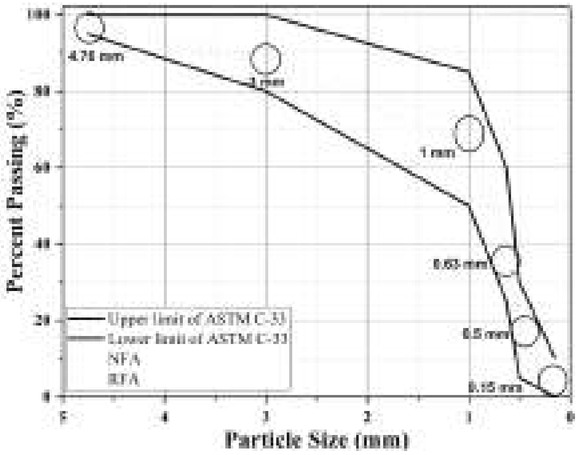

Fig. 1. Particle size of aggregates.

Table 1
Properties of the materials used.

Properties	Natural fine aggregate	Recycled fine aggregate	Cement	PVDF Nanofibers
Type	Natural	Recycled	Comercial	Polymeric
Fineness modulus	2.74	3.10	–	–
Water absorption (%)	4.35	13.22	–	–
Bulk density (kg/cm^3)	2.45	2.25	3.15	–
Humidity (%)	5.11	0.12	–	–
Averege size (cm)	0.08	0.075	0.0015–0.0025	0.0000788
Polymer concentration (%)	–	–	–	17
Mw (g/mol)	–	–	–	534,000

2.3. Synthesis and separation of PVDF NFs

A polymeric solution of 17 % PVDF in DMF and acetone, at a ratio of 70:30, was prepared. Subsequently, the solution was heated at 100 °C for 3 min and then stirred for 12 h. After this, the solution was placed in a 5-ml syringe for the electrospinning process. The infusion pump used was a NE-300 syringe pump with a high-voltage source (ES30P–5W/DAM Gamma high-voltage). The PVDF NFs were obtained at a flow rate of 0.5 ml/h, a distance of 18 cm, and a voltage of 22.5 kV.

Separation of the PVDF NFs was performed using a Polytron PT-2100 homogenizer. The process started by adding the PVDF NFs to a beaker (with the weight of the mixture design) along with distilled water (volume specified in the mixture design). The homogenizer was then placed inside the beaker and was set to 20,000 rpm for 8 min. After this time, the water in the mixture had turned white and no agglomerations of PVDF or NFs were visible; this was used to make the mortar mixture.

2.4. Mortar design

The mixture design of the recycled mortars is given in Table 2. The water/cement (w/c) ratio was 0.47 and the aggregate/cement ratio was 2.75, based on ASTM C109 [36]. The percentage substitution of RFA for natural fine aggregate (NFA) was 25 %, and the different percentages of the additions of PVDF were 0.025, 0.05, and 0.1 %. The dosage shown for each sample corresponded to a mortar volume of 256 cm^3. The NFA sample corresponded to a mortar with natural aggregates without nanofibers, the RFA sample was a mortar with 25 % recycled aggregates and no added NFs, and RFA-0.025 % corresponded to a mortar with 25 % recycled aggregates and 0.025 % PVDF NFs (similar nomenclature is shown for the 0.05 and 0.1 % additions of NFs).

Table 2
Mixture design.

Materials	Samples				
	NFA	RFA	RFA-0.025 %	RFA-0.05 %	RFA-0.1 %
NFA (g)	229.2	171.9	171.9	171.9	171.9
RFA (g)	0.000	57.3	57.3	57.3	57.3
Cement (g)	83.3	83.3	83.3	83.3	83.3
Water (ml)	39.2	39.2	39.2	39.2	39.2
Addition or reduction of water depending on humidity and absorption (ml).	−9.1	+14.6	+14.6	+14.6	+14.6
PVDF nanofibers (g)	0.0	0.0	0.021	0.042	0.084

2.5. Characterization and specimens

2.5.1. PVDF NFs

To analyze the morphology of the PVDF NFs, a FEI Nova Nano scanning electron microscope (SEM), working at a voltage of 5 kV, a working distance of 0.4–0.6 cm, and fitted with a vacuum detector, was used. The diameter of the NFs was measured using ImageJ software, taking the average of 100 measurements. The bond vibration of the PVDF NFs was analyzed by Fourier-transform infrared (FT–IR) spectroscopy using a Bruker Alpha II spectrometer, with a scanning range of 4000 to 500 cm^{-1}, a resolution of 4 cm^{-1}, and 16 scans. The thermal behavior of the PVDF NFs was analyzed by thermogravimetric/differential thermal calorimetry (TGA/DSC), using SDT Q600 TA Instruments equipment in a nitrogen atmosphere up to 700 °C, at a heating rate of 10 °C/min. The PVDF NFs were then analyzed by X-ray diffraction (XRD) using a Bruker D2 phaser, at 30 kV, in a range of 10–80 °2θ and at a counting time of 1 s/step.

2.5.2. Mortar specimens

The mortars were mixed according to the procedure described in ASTM C-305 [37]. The mortar flow test based on ASTM C 1437 [38] and the compressive and flexural strength were evaluated following the methodology established in ASTM C-348 [39] and ASTM C-349 [40], respectively. The properties were evaluated at 7, 14, and 28 days of curing (water-immersion curing). The total porosity was determined by vacuum saturation of 5 cm^3 specimens of mortar after 28 days of curing [41]. To evaluate the effective porosity of the mortar specimens, they were cut into discs 7 cm high and 7 cm in diameter at 28 days of curing (ASTM C-1585 [42]). The chloride-ion permeability test was performed according to ASTM C-1202 methodology [43] in mortar specimens 10 cm in diameter and 20 cm high. It was necessary to apply vacuum to the specimens at a pressure of 0.1 cm Hg for 3 h, then they were saturated in water for 18 ± 2 h. The specimens were then placed between two acrylic cells, one filled with 0.3 % sodium hydroxide (NaOH) aqueous solution (anode) and the other with 3 % sodium chloride (NaCl) aqueous solution (cathode). Measurements were taken by connecting the cells to a 10-V supply for 6 h (with the current recorded every 5 min).

3. Results and discussion

3.1. NFs

Fig. 2 gives the characterization of the PVDF NFs. Fig. 2(a and b) show the morphology of the PVDF NFs synthesized by the electrospinning technique, which have a well-defined structure, are continuous, not bonded together, have different diameters, and show glossy internal sections, which could indicate defects or voids. This NF morphology could promote greater adhesion, anchoring, and bonding between the NF and the cementitious matrix [44]. Other research has shown similar NF morphology [45]. The size distribution of the NFs is illustrated in Fig. 2c, which shows that the diameters ranged from 500 nm to 1200 nm, with the highest proportion lying between 600 and 800 nm. The average diameter was 788 nm (several authors have agreed that fibers with these dimensions are considered NFs) [46–48]).

The FT–IR spectra of the PVDF NFs are given in Fig. 2d. Vibration of the carbon–hydrogen (C–H) bonds was observed at 2960 and 841 cm^{-1}, which correspond to stretching and rocking vibrations, respectively [49]. Carbon–fluorine (C–F) bond-stretching vibrations were observed at 1401 and 881 cm^{-1}, with C–F out-of-plane vibrations at 1280 cm^{-1} [45]. The peak at 1187 cm^{-1} corresponded

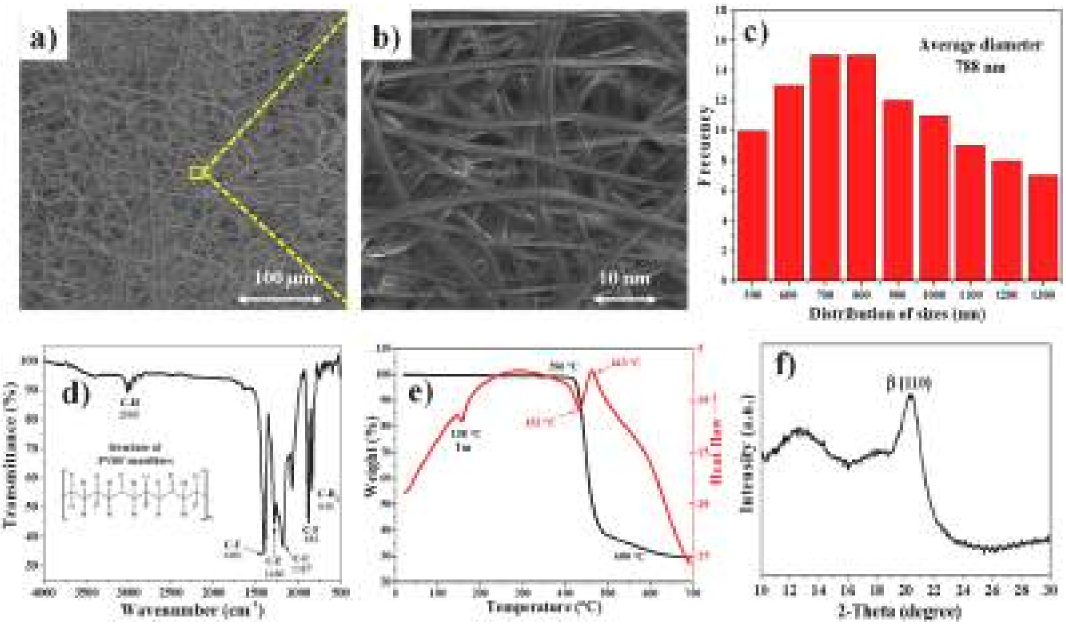

Fig. 2. Characteristics of NFs de PVDF: a y b) SEM, c) distribution of sizes, d) FT-IR, e) TGA/DSC and f) XRD.

to the stretching vibration of the carbon–carbon (C–C) bond [50,51]. This analysis showed there were no residues of the solvents used in the electrospinning process in the nanomaterial.

The results of the thermal analysis are provided in Fig. 2e, which shows two characteristic weight losses of the polymer. The first corresponded to the separation of the C–H and C–F bonds in the main chain, forming hydrogen fluoride (HF). This loss occurred from 410 to 480 °C (the maximum loss observed in the DSC at 432 °C) [52]. The second weight loss with respect to temperature occurred after the degradation of the previously formed HF molecules at 463 °C [53]. The melting temperature of the PVDF NFs, 150 °C, was in agreement with that reported in the literature [54]. Fig. 2f gives the results of the XRD study of the synthesized NFs. The characteristic peak of this material was at 20.3°, which corresponds to the (110) plane reflections of the β-phase of the polymer [55,56]. These characterizations confirm that the NFs synthesized by electrospinning were free of contaminants.

3.2. Mortars

3.2.1. Mortar flow

To study the flowability of the mortar, it was first necessary to make several test mixtures with different w/c ratios in order to determine the amount of water necessary for all the study samples to fall within the plastic consistency range of the cement mortar (see Fig. 3a). It could clearly be seen that, by increasing the w/c ratio in the mortars, the fluidity increased because there was a greater amount of water. Using a w/c ratio of 0.32–0.42 created mortars with a dry consistency, whereas by increasing the ratio from 0.42 to 0.47, the mortar was within the limits of plastic consistency. After performing a series of tests, it was determined that using a w/c ratio of 0.47 would bring all the study samples into the desired flow range. Fig. 3b shows the mortar flow values of all the studied samples. The highest value of mortar consistency was for Sample NFA. In addition, it was observed that the samples containing RAs had a lower percentage of flowability. This was due to the nature of the RA, being a less-dense material with high porosity. This causes it to absorb large amounts of water [57]. It is worth mentioning that the use of PVDF NFs caused a decrease in the flowability of the mortar (the higher the amount of NFs, the lower the flowability). This is because the NFs helped to obstruct the free movement of water in the mixture [15].

3.2.2. Total porosity

The porous nature of RFA means that, when used in cement mortars, it significantly increases their total porosity, and so its analysis is of great importance [58]. Fig. 4 shows the total porosity of the mixtures used here. It can be seen that the mixture containing RFA with no NFs had the highest porosity (18.38 %). This is due to this type of aggregate having a lower density and tending to absorb water from the mix, causing an unreacted surface, which results in higher porosity [59]. The increase in the porosity of this mixture relative to the reference mortar (NFA) was 4 %. It was found that the use of PVDF NFs at 0.025, 0.05, and 1 % helped to decrease the percentage of porosity in the mortar by 0.6, 1.4, and 3 %, respectively, compared with the RFA sample. These results show that PVDF NFs help to densify the cementitious matrix, filling existing pores, thereby decreasing the overall porosity. Importantly, they helped counteract the effect of using RFA.

3.2.3. Mechanical properties

It has been reported in the literature that the excessive use of RFAs in cementitious materials decreases their mechanical properties, but with some researchers agreeing that the replacement of 25 % of the RFAs by NFAs did not cause such significant changes [60]. It has also been stipulated that the use of NFs helps to reinforce the microstructure of cementitious materials, providing greater resistance to the internal stresses generated by mechanical tests [13].

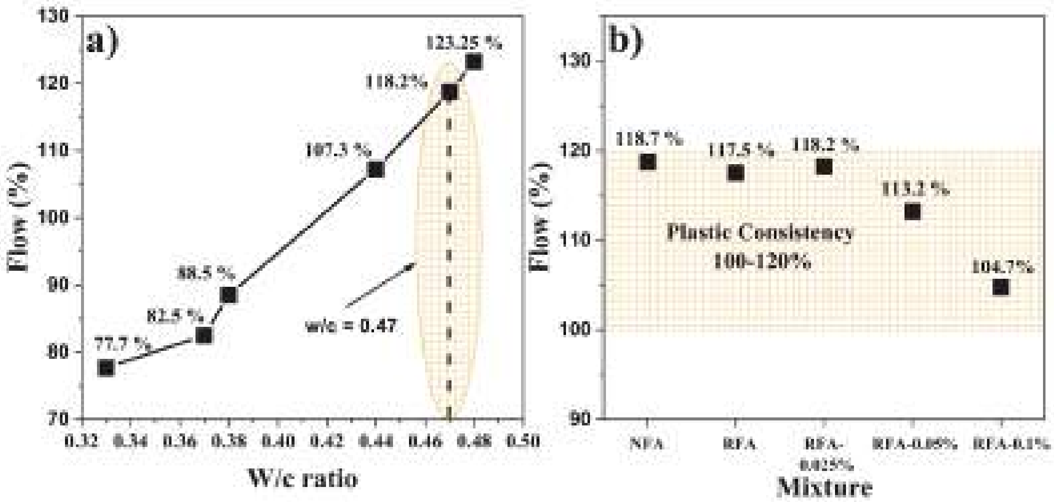

Fig. 3. a) Selecting the w/c ratio and b) Flow of mortars.

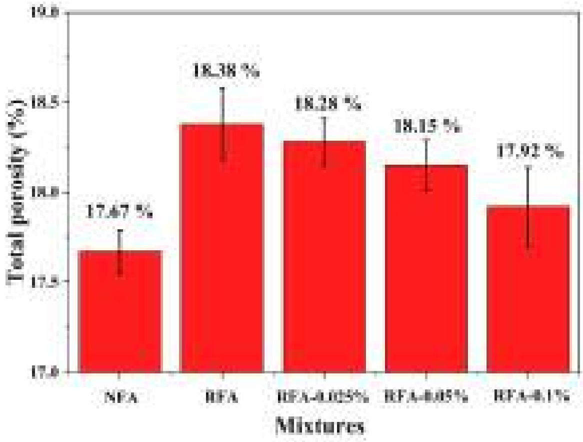

Fig. 4. Total porosity of mortars.

Fig. 5 provides the mechanical properties of the mortars produced for this study. In all the samples, as the days of curing increased, so did the mechanical properties in all the samples. This was to be expected because, at that age, cementitious materials attain their highest mechanical strength. At 28 days of curing, the RFA mix achieved a compressive strength very similar to that of the reference mortar (NFA), with a slight decrease of 0.6 %. This decrease is attributed to the nature of the RFA. The use of PVDF NFs in the mortars with RFAs promoted increases of 4.6, 11.3, and 12 %, with an addition of NFs of 0.025, 0.05, and 0.1 %, respectively, in compressive strength, compared to the RFA sample at 28 days of curing.

Regarding the flexural strength (Fig. 5b), the same trend was observed, with the RFA sample decreasing by 4.6 % with respect to the reference mortar (NFA). The use of PVDF NFs caused the mortar to increase in flexural strength by 7.3, 19.8, and 23.2 %, with additions of 0.025, 0.05, and 0.1 %, respectively, at 28 days of curing. The increase in the flexural strength of the mortars was more notable than the compressive strength results. This is due to the fact that, by their nature, the fibers work in tension and not so much in compression [61]. The effect of the NFs on the mechanical properties could include decreased porosity and densified cementitious matrix (from the total porosity study, Fig. 4), resistance to internally generated stresses, and help in stopping crack propagation through the seam effect [62].

The effect of the NFs on the mechanical properties can be viewed in Fig. 6. The main function of the fibers was in resisting tensile stresses. In the compression test, these stresses were distributed throughout the cementitious matrix [63]. At these sites, the NFs were able to absorb the stresses and increase the compressive properties (although these were not so significant increases). When a mortar is subjected to flexural stresses, most of the stresses are located at the site opposite to that where the load was applied, leading to crack generation and propagation, and fracturing of the material [64]. In the mortars containing NFs, the stresses generated by the bending stress were supported by the NFs dispersed in the cementitious matrix, preventing crack propagation, decreasing the fracture rate, absorbing the stress energy, and densifying the matrix. This greatly increased the mechanical properties of the mortars.

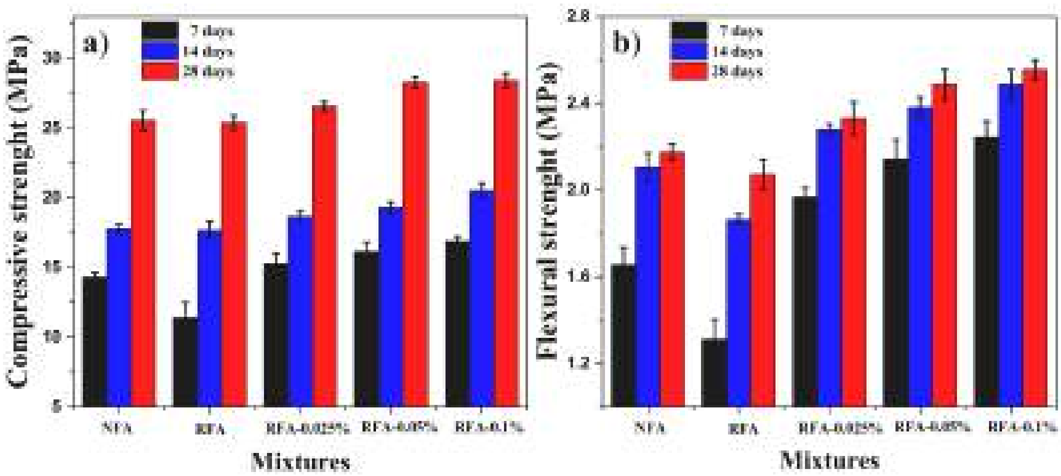

Fig. 5. Properties mechanical or mortars, a) compressive strenght and b) flexural strength.

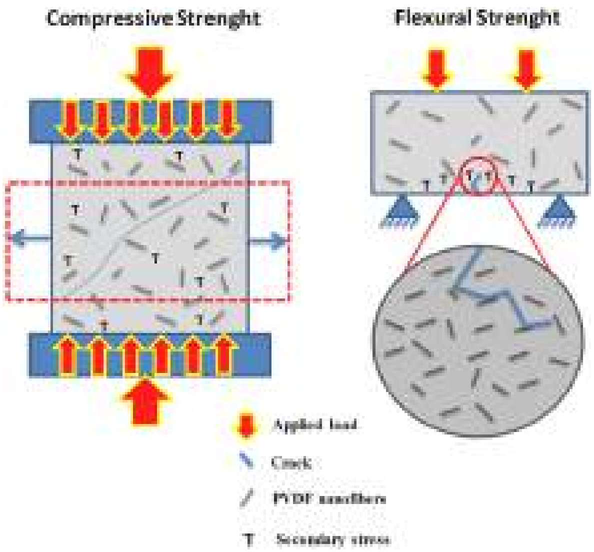

Fig. 6. Effect of PVDF NFs on mechanical properties.

3.2.4. Chloride-ion permeability

One of the most important durability analyses performed on the cementitious materials containing RFAs (due to their high porosity) was the permeability of the chloride ion because this ion is responsible for corroding and deteriorating concrete structures when associated with reinforcing steel [65].

Fig. 7 shows that all the samples had low chloride-ion permeability. The NFA and RFA mortars had through-loading values below 1000 C, indicating that the values were below the "very low" category based on the accelerated chloride-ion penetration classification (ASTM C1202) [66]. Recycled mortars containing PVDF NFs saw an increase in the permeability of the chloride ion (step charge values of between 1000 and 1200 C). This is probably due to PVDF having piezoelectric properties; when in contact with any electric field, it can be excited, serving as a bridge for the chloride-ion flow. The increase was 15, 20, and 21 % for the 0.025, 0.05, and 0.1 % additions, respectively, compared to the RFA sample. A possible mechanism for the ion transfer in the mortar microstructure by the PVDF NFs can be seen in Fig. 7.

3.2.5. Mortar microstructure

The microstructure of the recycled mortar reinforced with PVDF NFs is illustrated in Fig. 8. There was a physical interaction between the cement hydration products (e.g., hydrated calcium silicate [CSH], portlandite, and ettringite) and the PVDF NFs. In addition, no repulsion was observed between the mentioned components, and the nanomaterial was not agglomerated. This indicates a good mixing and dispersion process. At the interface between the NFs and the hydration products, the porosity was not high because the NFs were embedded in the cement paste [see Fig. 8(a, b, and d)], and thus they helped to improve the mechanical properties and durability of the mortar. To corroborate the presence of the nanomaterial in the recycled mortar, energy-dispersive spectroscopy was performed [Fig. 8c], which indicated elements such as calcium (Ca), oxygen (O), silicon (Si), and aluminum (Al), which correspond to

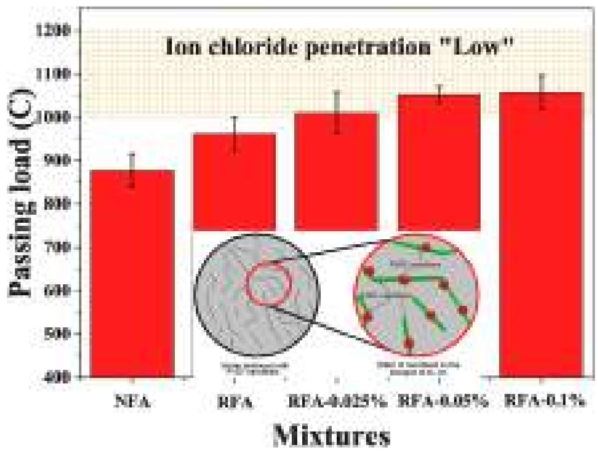

Fig. 7. Permeability of Cl^{-} ion in mortars.

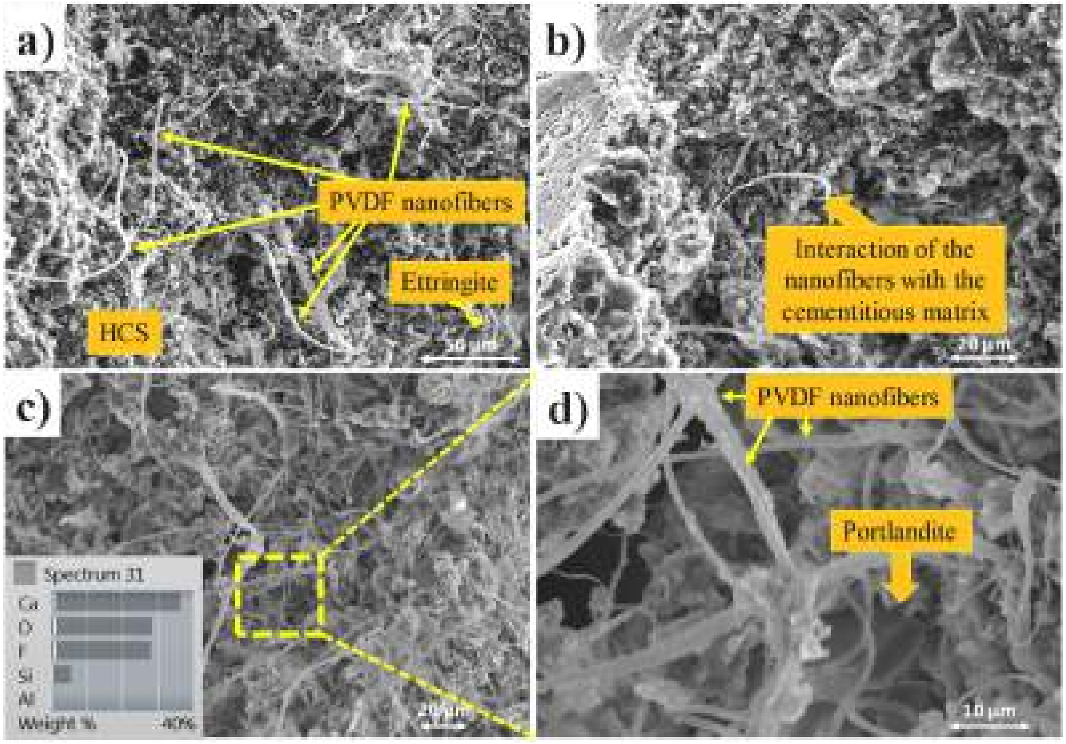

Fig. 8. Interaction of PVDF NFs in the mortar microstructure.

the nature of the hydration products. Fluorine was also detected in this analysis, confirming the presence of the nanomaterial in the microstructure of the recycled mortar. The presence of PVDF nanofibers in the recycled mortars caused a decrease in the fluidity of the mortar, a decrease in porosity, increases in resistance to compression and flexion and an increase in the transfer of the Cl^- ion. These results are attributed because these nanofibers provide the mortar with additional tensile strength and prevent the propagation of cracks, causing increases in mechanical properties [67,68]. In addition, they help to fill cavities or pores founded in the microstructure, causing a decrease in its porosity [69,70]. They also have the characteristic of retaining water from the mixture through physical iterations, which causes the fluidity of the mortar to decrease [71] and finally, being a piezoelectric material, it causes a greater transfer of the Cl^- ion. Therefore, the addition of this nanomaterial is beneficial to increase the applications of recycled mortar and contribute to sustainable construction.

4. Conclusions

1. The use of RAs in civil engineering is a significant advance toward sustainability in construction. For this reason, new alternatives must be founded to help counteract the negative effects caused by their use.
2. The electrospinning method was effective in generating PVDF nanofibers. The nanofibers obtained had an average diameter of 788 nm, they are separated from each other and had a smooth uniform morphology. Furthermore, the nanofibers were shown to have high thermal properties and high purity. With this characterization it was demonstrated that the electrospinning technique is effective and could be considered for its use in the construction industry.
3. Our findings show that the use of polymeric NFs in recycled mortars resulted in improved properties even better than those in conventional mortars. The interaction between the PVDF NFs and the hydration products was beneficial, causing decreases of up to 3 % in the total porosity of the samples. In addition, in the mechanical properties of compression and bending they caused to increase by 12 and 23.2 %, respectively.
4. It was also found that the penetration permeability of chloride ions increased with the addition of the PVDF NFs due to the piezoelectric nature of the material. This opens up a possibility of future research concerning their use in cementitious materials for creating conductive or piezoelectric mortars, or mortars with electronic properties.
5. The results of this research show that small proportions of nanomaterials can be used in mortars to help improve the characteristics of cementitious materials and that there is potential for new applications.

CRediT authorship contribution statement

M.J. Chinchillas-Chinchillas: Conceptualization, Investigation, Writing – original draft. **H. Cortez-Rodríguez:** Data curation, Methodology, Software. **G.J. Fajardo-San Miguel:** Investigation, Resources. **M.J. Pellegrini-Cervantes:** Conceptualization, Supervision, Writing – review & editing. **M. Rodriguez-Rodriguez:** Project administration, Supervision, Visualization. **R. Corral-Higuera:** Methodology, Validation. **S.P. Arredondo-Rea:** Investigation, Visualization. **Alberto Gaxiola:** Resources, Software. **A. Castro-Beltrán:** Formal analysis, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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