

Research Paper

Green Fibers-Reinforced Cement Mortar with the Inclusion of Nano-CaCO₃ and Metakaolin

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Over the previous few decades, there has been a noticeable increase in interest in the use of vegetable fibers and supplemental cementitious elements in mortar and concrete. The date palm frond was utilized in this study to create date palm fibers (DPF), which were then added to the cement mortar at percentages of 1%, 2%, 3%, 4%, and 5% by cement weight. There were two types of DPFs used: one type was untreated, and the other had a mechanical treatment that created holes before applying a layer of polychloroprene (neoprene) on the surface. Metakaolin (MK) and nano calcium carbonate (nano-CaCO₃) were added to the cement mortar by the weight of cement. MK was replaced by 10% of the weight of cement. Besides, the nano-CaCO₃ was replaced by 1%, 2%, 3%, and 4% of the weight of cement. Mechanical tests for flowability, compressive strength, and flexural strength were conducted. In addition, one MCDM methodology called VIKOR is utilized to choose the best combination out of several combinations and criteria. The results indicate that a higher DPF concentration enhances both compressive and flexural strength. The mixtures with the DPF coating and mechanical treatment give the strongest and most significant results. In addition, the flowability of cement mortar decreases when the DPF concentration increases. In addition to the high content of nano-CaCO₃ in cement mortar, given the greater reading of strength, the presence of nano-CaCO₃ in cement mortar reduces the disparity in result values that have a higher DPF content. The mixtures containing 4% and 5% DPF and 3% and 4% nano-CaCO₃ are the optimal ones, according to the VIKOR technique.

Keywords: fibers reinforced cement mortar; date palm fibers (DPF); nano calcium carbonate (nano-CaCO₃), metakaolin (MK); pozzolanic materials.

1. INTRODUCTION

There has been a significant surge of interest in the incorporation of supplementary cementitious materials such as silica fume, fly ash, and metakaolin

in concrete based on ordinary Portland cement (OPC) over the past few decades [1–7]. The utilization of this technology presents an effective approach to reducing the carbon footprint of concrete by minimizing the amount of ordinary OPC used. Incorporating supplementary cementitious materials (SCMs) in place of OPC offers potential benefits, including improved mechanical properties and durability [8–13]. Metakaolin, a representative supplementary cementitious material derived from controlled calcination of kaolin clay, exhibits exceptional pozzolanic reactivity [14]. Partially replacing OPC with metakaolin in concrete leads to significant improvements in the mechanical properties. This is due to the increased production of binding material, calcium silicate hydrates (C-S-H), through the pozzolanic reaction between metakaolin and calcium hydroxide (CH). Furthermore, the extensive surface area of metakaolin provides more sites for OPC hydration, enhancing the overall hydration process [15]. While ground limestone is commonly considered an inert mineral filler due to its composition, which mainly consists of calcite (CaCO_3) and particle sizes similar to regular OPC [16], adding a significant amount of pulverized limestone to concrete can significantly reduce its strength due to dilution. However, incorporating a small quantity of crushed limestone can slightly increase the compressive strength of concrete, as the reactivity of limestone powders with the aluminate phases in OPC is limited [17, 18]. This reaction between aluminate phases and calcium carbonate in limestone produces and stabilizes ettringite, a hydration product with a larger volume compared to other OPC hydration products. As a result, the microstructure of the hardened concrete becomes denser, leading to higher compressive strength. Antoni *et al.* demonstrated that leveraging the synergistic effects between ground limestone and metakaolin can further improve the properties of metakaolin-blended cement-based concrete [19–22]. The inclusion of CaCO_3 can influence the distribution of lime, alumina, and sulfate within the concrete, thereby altering the mineralogy of the hydrated cement pastes [23, 24].

According to research by BALASUBRAMANIAN and SELVAN [25], the utilization of natural vegetable fibers in cement composites offers various advantages compared to fiberglass-reinforced components. These advantages include a 10% reduction in weight, an 80% reduction in energy consumption during production, and a 5% reduction in component costs. However, incorporating natural vegetable fibers into concrete has also been associated with certain disadvantages.

In 2018, ÇOMAK *et al.* [26], determined that cement mortars reinforced with 2–3% amount and 12 mm length of natural hemp fiber give the optimum results, improving significantly performance in compressive strength, flexural strength, and splitting tensile strength. Besides, the effect of hemp fibers on the flow of fresh concrete is almost negligible.

Most natural vegetable fibers have a high water absorption capacity, which can lead to poor workability in fresh concrete and degradation in alkaline environments. Consequently, these factors can negatively affect desirable properties such as tensile strength and bond strength [27–33]. Nevertheless, researchers have proposed various treatments for these fibers, showing the potential to overcome these drawbacks [34–39]. Additionally, it has been argued that the high water absorption of natural vegetable fibers can be advantageous for the internal curing of the composite [40]. According to research by LERTWATTANARUK and SUNTIJITTO [41], the use of natural vegetable fiber-reinforced cement composites instead of asbestos cement composites offers significant advantages in terms of non-toxicity. This substitution eliminates the risks associated with human exposure to diseases such as asbestosis, cancer, malignant pleural disease, and tumors. In response to these health concerns, several countries have implemented legislation against the use of asbestos [42]. The study also emphasizes that the process of obtaining natural vegetable fibers is environmentally sustainable and free from pollution. Furthermore, since these fibers are widely available worldwide, they can be locally enhanced to suit specific climate requirements, reducing the need for material imports [43].

The aim of this work is to effectively create a sustainable cement mortar employing natural fibers (DPF). The research also emphasizes the use of modified and treated palm fronds. Furthermore, metakaolin was substituted by cement weight, and the pozzolanic material, represented by nano- CaCO_3 , was used. The mechanical characteristics (compressive strength and flexural strength) of cement mortar reinforced with green fibers were investigated.

2. EXPERIMENTAL WORK

2.1. *Materials*

Samples were prepared using a general-purpose cement-OPC, manufactured by Tasluga/Iraq. The chemical and physical analyses of the OPC are shown in Tables 1 and 2. Sample preparation process was conducted following ASTM C150 [44]. The fine aggregate (sand) used in this study was sourced from the Al-Akhdar Region in Karbala. Furthermore, the sieve analysis of the fine aggregate was conducted in accordance with ASTM C33 [45] and is presented in Table 3. MK was supplied from the Dwekhla region of Iraq, and Table 4 shows the composition of MK. Nano- CaCO_3 is added to the mixture as an additional ingredient in powder form, replacing the weight of cement with particles approximately 10–45 nm in size. The physical characteristics of nano- CaCO_3 are shown in Table 5.

Table 1. Chemical properties of Portland cement.

Composition	Content [%]	Specification ASTM C150 [44]
CaO	64.0	–
SiO ₂	21.0	–
Al ₂ O ₃	5.00	–
Fe ₂ O ₃	2.60	–
MgO	2.22	<6%
SO ₃	2.38	<3%
L.O.I.	3.25	<3%
Insoluble residue	1.1	≤0.75%
Lime saturation factor, L.S.F.	0.95	0.66–1.02
Main compounds (Bogue's equations)		
C ₃ S	57.50	–
C ₂ S	14.30	–
C ₃ A	8.70	–
C ₄ AF	10.80	–

Table 2. Physical properties of Portland cement.

Physical properties		
Test	Results	ASTM C150 [44]
Initial setting time [min]	119 295	Not less than 45 min Not more than 375 min
Fineness (Blaine) [m ² /kg]	485	Min. 280 m ² /kg
Compressive strength of 50 mm cubic mortar specimen [MPa] 3 days 7 days	22.5 25.0	Min. 12 MPa Min. 19 MPa

Table 3. Sieve analysis of sand.

Sieve size [mm]	Passing [%]	Passing [%], ASTM C33 [45]
4.75	93	90–100
2.36	86	85–100
1.18	82	75–100
0.60	70	60–79
0.30	30	12–40
0.15	7	0–10

Table 4. Composition of MK.

Compound	Percentage by mass
SiO ₂	52–53
Al ₂ O ₃	42–43
CaO	0.02–0.1
Fe ₂ O ₃	0.5–1
MgO	0–1.0
SO ₃	0–0.1
Na ₂ O	0–0.05
K ₂ O	0.4–1.5

Table 5. Physical properties of nano-CaCO₃.

Properties	Values
Morphology	Cubic or hexagonal
Color	White
pH	Not applicable
Bulk density [g/mL]	0.68
True density [g/cm ³]	2.9
Specific surface area [g/m ²]	30–60
Average particle size [nm]	10–45 nm
Purity [%]	97
Melting point	825
Boiling point [°C]	Decomposes
Molecular weight [g/mol]	100.09

2.2. Preparation of palm frond fibers

DPFs are used as natural fibers sourced from palm trees. The properties of DPF are shown in Table 6. The frond palm is cut into many sizes, about 8–18 mm in length. The DPF was used in two ways: the first without any treatment of

Table 6. Properties of DPF.

Properties	Values
Tensile strength [MPa]	280 ± 60
Density [g/cm ³]	0.25–1.1
Length [mm]	8–18
Diameter [μm]	100–800

the DPF, and the second included a mechanical treatment where voids or holes were created in the DPF. The resulting fibers were placed in an oven at 80°C for 48 hours. Figure 1 demonstrates the mechanism of bonding between DPF and cement mortar.

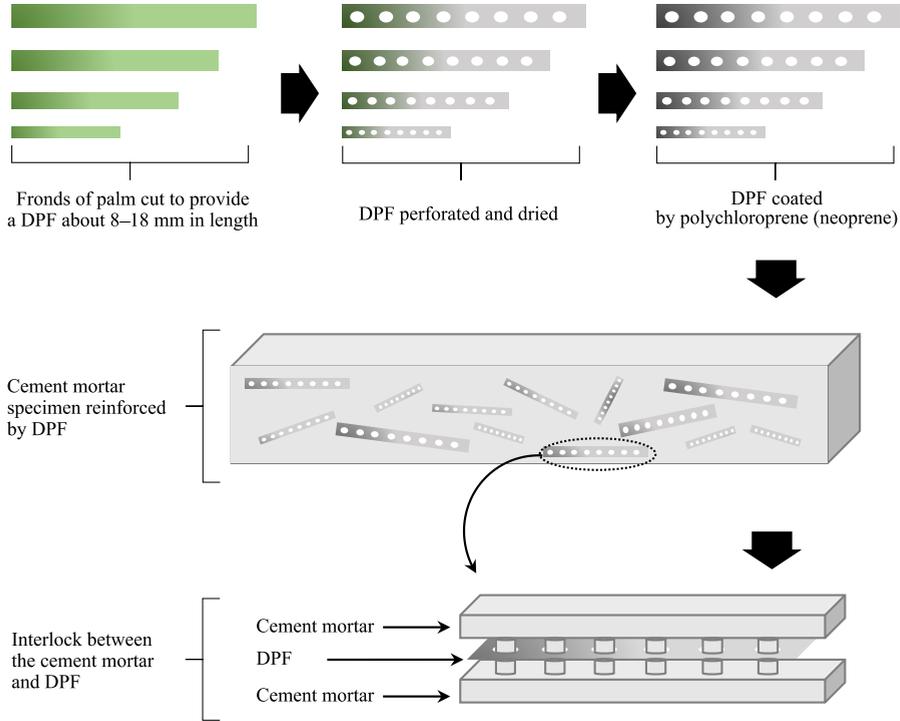


FIG. 1. The mechanism of bonding between DPF and cement mortar.

The DPF was coated or surface-treated with polychloroprene (neoprene), applied to the DPF surface as shown in Fig. 2. This rubber has a good balance of mechanical properties and fatigue resistance, which is second only to that of natural rubber but has superior oil, chemical, and heat resistance. Polychloroprene

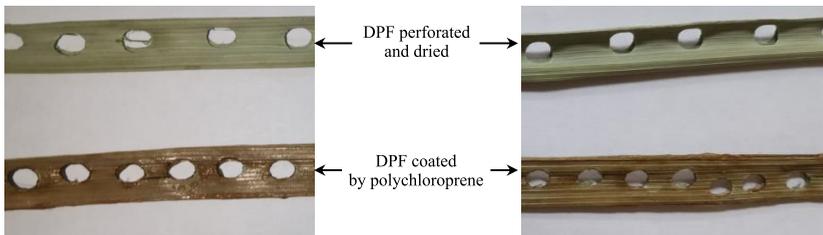


FIG. 2. The DPF perforated and coated surface by polychloroprene (neoprene).

exhibits excellent resistance to heat, ozone, weathering, and chemicals, making it highly durable. It also maintains flexibility over a wide temperature range, including both low and high temperatures. These properties make polychloroprene well-suited for various industrial and commercial applications. Neoprene is commonly used in the production of protective coatings, adhesives, gaskets, seals, and various types of rubber products. It is a popular choice in various industries due to its durability, flexibility, and chemical resistance, making it a preferred material in the production of wetsuits, gloves, and other items requiring water and abrasion resistance.

2.3. Mixing and preparation of specimens

The mix proportion was 1:1, 0.45 (cement: sand, w/c = 0.45). Different fiber contents were added to the mortar mix. The DPF content was set at 1%, 2%, 3%, 4%, and 5% by weight of cement, as shown in Table 7. Cube molds of $50 \times 50 \times 50$ mm were cast for compressive strength testing in accordance with ASTM C109 [46]. Flexural strength tests were performed on $40 \times 40 \times 160$ mm beams subjected to the three-point flexural test in accordance with ASTM C348 [47]. A flow table test was performed to assess the workability of the mortar according to ASTM 1437 [48]. According to ASTM C192 [49], the specimen was stripped after 24 hours of casting and immersed in normal water at a limiting temperature of roughly $23 \pm 2^\circ\text{C}$.

3. RESULTS AND DISCUSSION

3.1. Flowability

The flowability of cement mortar reinforced by DPF, including MK and different contents of nano- CaCO_3 is assessed. The results of cement mortar reinforced with DPF are presented in Table 8. The control mix (M0) without DPF, MK, and nano- CaCO_3 revealed a flow value of 150%. Generally, the results exhibit a reduction in flowability with an increase in DPF content. This is attributed to the fact that the cement mortar matrix contained DPF that was dispersed randomly, worked as a skeleton, and finally hindered the flow of the cement mortar mixture [50–53]. Figure 3 shows the relationship between DPF content and flowability.

The lowest value of flow was 117% for mix M10 compared to the control mix. Alongside, it can be observed that the mixes containing coated DPF show higher flowability compared to the same mixes containing uncoated DPF. This is attributed to the smoother surface of DPF coated with neoprene, resulting in reduced friction with the cement mortar matrix. Additionally, nano- CaCO_3 and MK have an effect on flowability, which is contributed to higher surface

Table 7. Mix properties.

Mixes	Mix proportions	DPF ^a [%]	MK ^b [%]	Nano-CaCO ₃ ^c [%]	Specimen no. of compressive strength		Specimen no. of flexural strength	Total no. of test specimens
					7 days	28 days		
Control mix	M0	0	0	0	3	3	3	12
	M1	1	10	0	3	3	3	12
Without treatment	M2	2	10	1	3	3	3	12
	M3	3	10	2	3	3	3	12
	M4	4	10	3	3	3	3	12
	M5	5	10	4	3	3	3	12
	M6	1	10	0	3	3	3	12
Mechanical treatment	M7	2	10	1	3	3	3	12
	M8	3	10	2	3	3	3	12
	M9	4	10	3	3	3	3	12
	M10	5	10	4	3	3	3	12
	M11	1	10	0	3	3	3	12
Without treatment	M12	2	10	1	3	3	3	12
	M13	3	10	2	3	3	3	12
	M14	4	10	3	3	3	3	12
	M15	5	10	4	3	3	3	12
	M16	1	10	0	3	3	3	12
Coated by polychloroprene (neoprene)	M17	2	10	1	3	3	3	12
	M18	3	10	2	3	3	3	12
	M19	4	10	3	3	3	3	12
	M20	5	10	4	3	3	3	12
	Total no. of specimens							

^a DPF added by weight of cement, ^b MK replaced by weight of cement, ^c Nano-CaCO₃ replaced by weight of cement.

Table 8. Results of the tests.

Mixes	Mix proportions	DPF ^a [%]	MK ^b [%]	Nano-CaCO ₃ ^c [%]	Compressive strength [MPa]		Flexural strength [MPa]		Flowability [%]	
					7 days	28 days	7 days	28 days		
Control mix		M0	0	0	24	32	4.5	6	150	
		M1	1	10	28	37	5.6	7.4	148	
		M2	2	10	30	40	6	8	143	
		M3	3	10	2	31	42	6.3	8.4	133
		M4	4	10	3	31	42	6.3	8.4	125
Uncoated		M5	5	10	4	30	41	6.2	8.2	120
		M6	1	10	0	28	38	5.7	7.6	145
		M7	2	10	1	32	43	6.5	8.6	140
		M8	3	10	2	33	45	6.8	9	138
		M9	4	10	3	33	44	6.6	8.9	126
Coated by polychloroprene (neoprene)	1:1, 0.45 (cement:sand, w/c = 0.45)	M10	5	10	4	33	44	6.6	9	117
		M11	1	10	0	30	40	6	8	150
		M12	2	10	1	31	42	6.3	8.5	145
		M13	3	10	2	33	45	6.8	9.1	138
		M14	4	10	3	33	44	6.6	9.2	130
		M15	5	10	4	35	47	7.1	9.4	125
		M16	1	10	0	31	42	6.3	8.4	145
		M17	2	10	1	33	44	6.6	8.9	142
		M18	3	10	2	33	45	6.8	9.3	136
		M19	4	10	3	35	47	7.1	9.6	127
M20	5	10	4	37	50	7.6	10	120		
Mechanical treatment		M20	5	10	4	37	50	7.6	10	120

^a DPF added by weight of cement, ^b MK replaced by weight of cement, ^c Nano-CaCO₃ replaced by weight of cement.

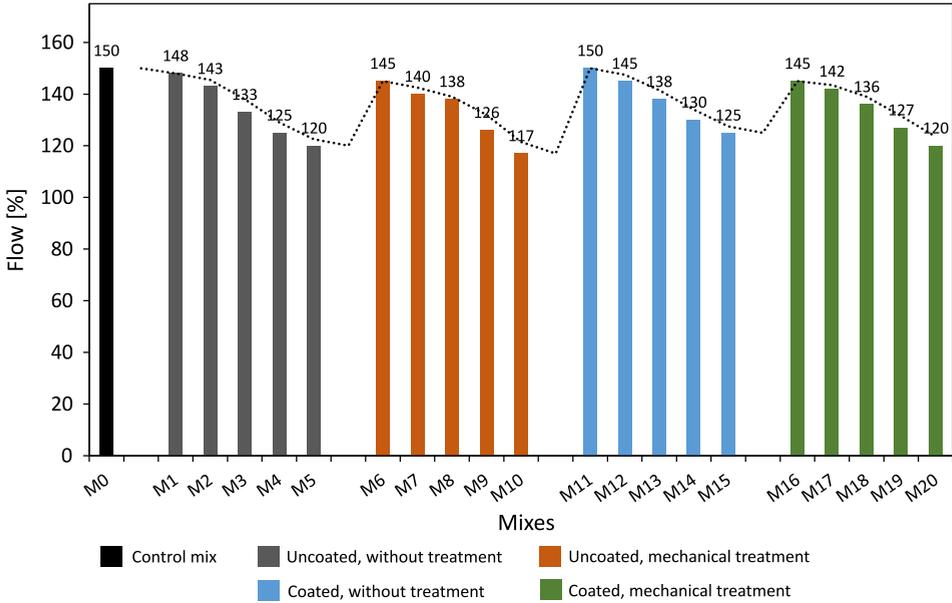


FIG. 3. Effect of DPF content on the flowability of cement mortar.

areas and finer particle sizes. Nanomaterials tend to absorb more water [54, 55], further increasing the cohesiveness of the cement mortar.

3.2. Compressive strength

The compressive strength of cement mortar reinforced by DPF with different volume fractions of fibers and inclusion of MK and nano- CaCO_3 is presented in Table 8.

Figure 4 shows the effect of DPF on the compressive strength of cement mortar. It can be observed that the compressive strength is significantly influenced by the addition of DPF. The compressive strength increases with the rising content of DPF, and this increasing is attributed to the uniform distribution of DPF in the cement matrix [56, 57].

In Fig. 5, the mixes with perforated DPF exhibit significant results compared to others mixes. The highest increase in compressive strength is observed in mixes M19 and M20, containing 4% and 5% mechanically treated and coated DPF. Additionally, it can be observed the mixes with MK and high nano- CaCO_3 content show higher compressive strength. This is attributed to the increased contribution of hydration products, leading to increased compressive strength due to nano- CaCO_3 , which in turn accelerates the C3A's reaction rate to produce carbon-aluminate [58–60].

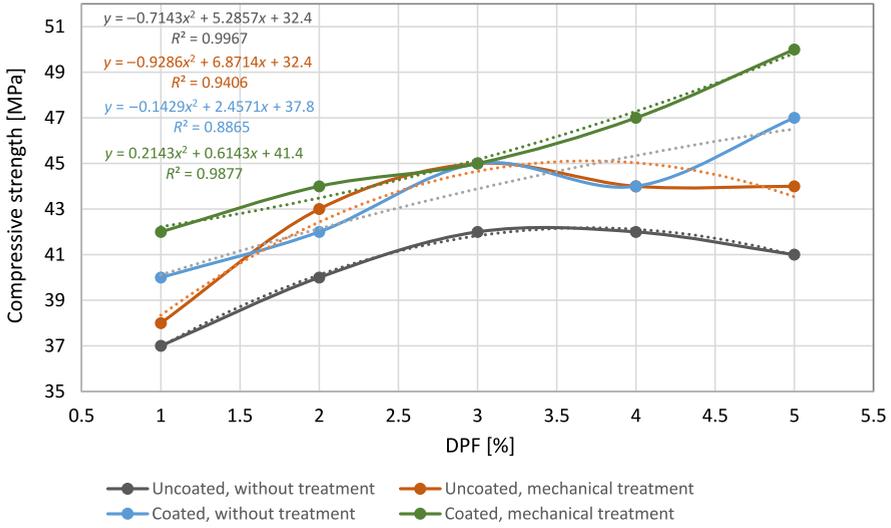


FIG. 4. Relationship between DPF content and compressive strength.

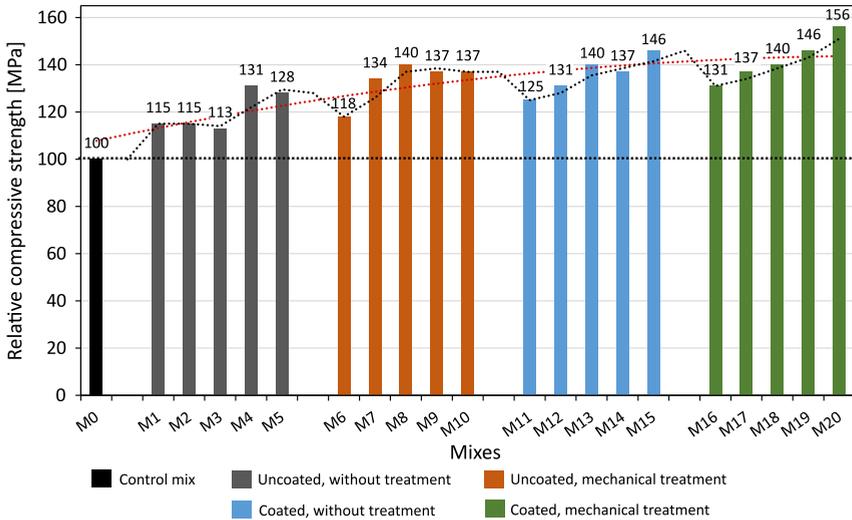


FIG. 5. Relative compressive strength of DPF reinforced cement mortar content.

3.3. Flexural strength

The flexural strength of cement mortar reinforced by DPF with different volume fractions of fibers and the inclusion of MK and nano-CaCO₃ are presented in Table 8.

Figure 6 shows the impact of DPF on the flexural strength of cement mortar. Flexural may be seen to have a major impact when the DPF is included. An in-

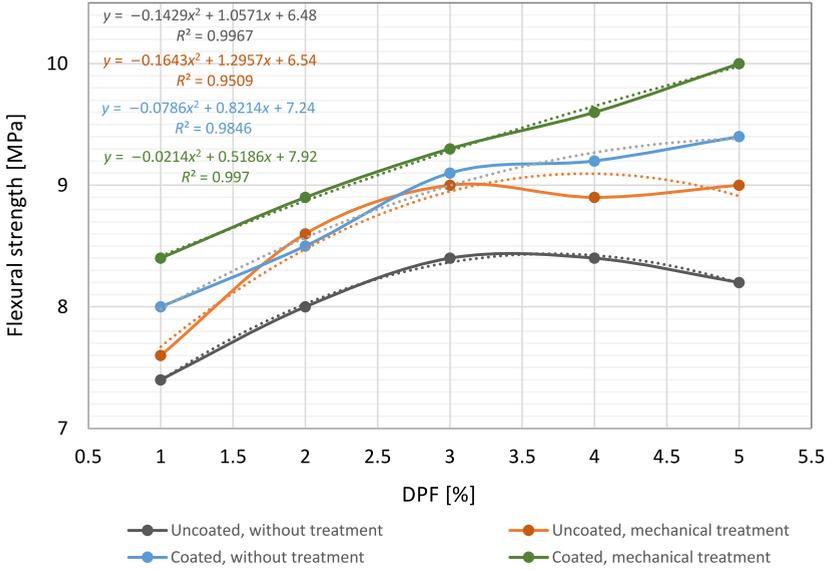


FIG. 6. Relationship between the flexural strength and DPF content.

crease in DPF concentration corresponds to an increase in flexural strength, attributed to the consistent distribution of DPF in the cement matrix [56, 57].

When compared to other mixtures, the mixes with perforated DPF exhibit interesting results. Figure 7 illustrates the increased flexural strength, partic-

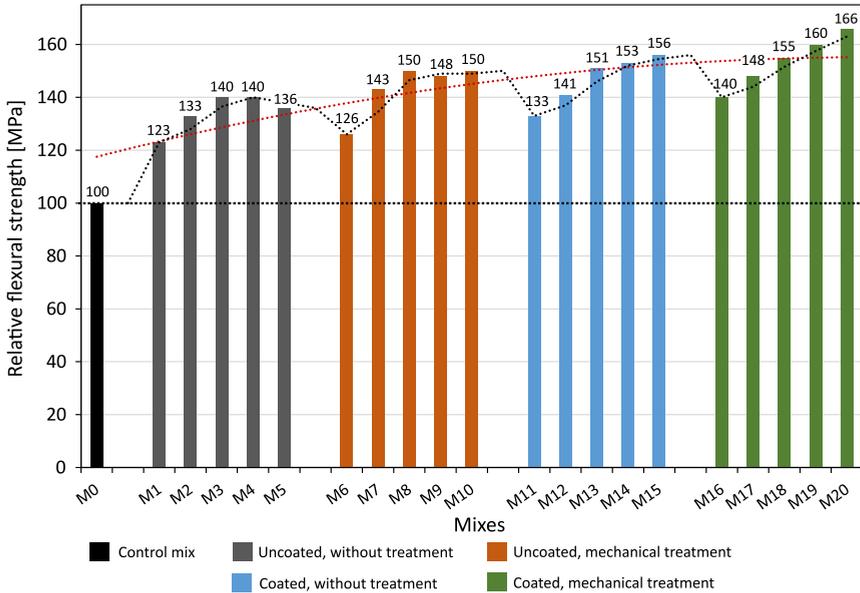


FIG. 7. Relative flexural strength of DPF-reinforced cement mortar content.

ularly notable in mixes M19 and M20, containing 4% and 5% mechanically treated and coated DPF. In addition, it is observed that mixes with high nano- CaCO_3 and MK content exhibit higher flexural strengths. This can be attributed to increased contributions from hydration products, which in turn boost strength due to nano- CaCO_3 , which accelerates the rate at which C3A reacts to produce carbon-aluminate [58–62]. Figure 8 illustrates the mechanism of the bonding between the fibers and mortar, where the interlock between the mortar and the perforated DPF is apparent, showing the failure mode after applying flexural strength.



FIG. 8. Mechanism of bonding between the DPF and cement mortar (a), and the failure mode after applying flexural strength (b).

3.4. *Vlsekriterijumska Optimizacija I Kompromisno Resenje (VIKOR)*

One of the multiple criteria decision making (MCDM) techniques is VIKOR (in English: *Multicriteria Optimization and Compromise Solution*) that evalu-

ates options based on their proximity to the best possible solution and distance from the worst possible solution or negative ideal solution. The VIKOR technique involves multicriteria optimization of complex systems, with an emphasis on choosing and prioritizing options from a range of options when competing criteria are present. VIKOR determines a multicriteria ranking index based on a specific measure of distance of each option to the optimal solution [63–65].

In this research, VIKOR was employed to find the optimal combination among various mixtures and criteria, considering factors such as needed strength, DPF fibers, and flowability. Conversely, the objective was to minimize the cost, density, certain fiber types, and cement content. Two types of criteria were considered: non-beneficial (minimum values are selected) and beneficial (maximum values that meet requirements are desired) [66–70]. The mathematical steps of VIKOR are outlined below:

Step 1: Find the best and worst values:

$$x_i^+ = \max x_{ij},$$

$$x_i^- = \min x_{ij},$$

where $i = 1, 2, 3, \dots, m$; $j = 1, 2, 3, \dots, n$.

Step 2: Normalization of S_j and R_j :

$$S_j = \sum \left[\frac{w_i(x_i^+ - x_{ij})}{x_i^+ - x_i^-} \right],$$

$$R_j = \max \left[\frac{w_i(x_i^+ - x_{ij})}{x_i^+ - x_i^-} \right],$$

where w_i is the weight of each criteria.

Step 3: Computation of Q_j :

$$Q_j = \frac{v(S_j - S^+)}{S_i^+ - S_i^-} + (1 - v) \left(\frac{R_j - R^+}{R^- - R^+} \right),$$

where the value of v is usually equal to 0.5.

Step 4: Sort the values of Q_j so that the lowest value, which represents a compromise, is the best option. This implies that a Q_j rating value can be positioned higher the lower it is.

According to the results in Table 8, the candidate results are shown in Table 9, determining the best and worst values along with assigned weights for each criterion.

Table 9. Candidate results.

Weight	0.15	0.15	0.25	0.25	0.2
Mix no.	DPF	Nano-CaCO ₃	Compressive strength	Flexural strength	Flowability
M0	0	0	32	6	150
M1	1	0	37	7.4	148
M2	2	1	40	8	143
M3	3	2	42	8.4	133
M4	4	3	42	8.4	125
M5	5	4	41	8.2	120
M6	1	0	38	7.6	145
M7	2	1	43	8.6	140
M8	3	2	45	9	138
M9	4	3	44	8.9	126
M10	5	4	44	9	117
M11	1	0	40	8	150
M12	2	1	42	8.5	145
M13	3	2	45	9.1	138
M14	4	3	44	9.2	130
M15	5	4	47	9.4	125
M16	1	0	42	8.4	145
M17	2	1	44	8.9	142
M18	3	2	45	9.3	136
M19	4	3	47	9.6	127
M20	5	4	50	10	120
Best+	5	4	50	10	117
Worst-	0	0	32	6	150

Calculating S_j , R_j and Q_j and ranking the alternatives to obtain the optimal mixes are shown in Table 10.

The outcomes displayed in Table 10 are derived using the VIKOR method computation, where mix M20, which has the lowest score of 0, is ranked as the top option. Moreover, mix M15 is placed second.

4. CONCLUSION

According to the test results, several conclusions can be drawn:

- 1) Cement mortar's flowability decreases when DPF content rises. Additionally, mechanical treatment of DPF resulted in the lowest flowability rating.

Table 10. Determining S_j , R_j and Q_j .

Mix no.	S_j	R_j	Q_j	Rank of mixes
M0	0.85	0.2	1	21
M1	0.685354	0.15	0.767424	20
M2	0.531793	0.118182	0.590255	15
M3	0.376616	0.088889	0.418896	12
M4	0.272753	0.088889	0.356803	8
M5	0.203636	0.1	0.345294	7
M6	0.650606	0.15	0.746651	19
M7	0.454823	0.1125	0.528997	14
M8	0.33601	0.095455	0.412236	11
M9	0.230076	0.066667	0.27167	5
M10	0.116667	0.066667	0.203871	4
M11	0.631111	0.15	0.734996	18
M12	0.493662	0.127273	0.59185	16
M13	0.33101	0.095455	0.409246	10
M14	0.233258	0.066667	0.273572	6
M15	0.099697	0.036364	0.112425	2
M16	0.566162	0.15	0.696168	17
M17	0.437803	0.113636	0.521871	13
M18	0.311919	0.086364	0.373443	9
M19	0.166288	0.045455	0.176625	3
M20	0.013636	0.013636	0	1
S+, R+	0.013636	0.013636		
S-, R-	0.85	0.2		

- 2) The compressive and flexural strengths are enhanced for mixtures with higher DPF concentrations. Given the significant results as compared to the control mix and other mixes, the mixes that employed mechanical DPF treatment were particularly noteworthy.
- 3) The addition of polychloroprene (neoprene)-coated DPF leads to a substantial increase in compressive and flexural strengths, with Mix M20 (containing approximately 5% DPF) showing an improvement of around 56% and 66%, respectively, compared to the control mix.
- 4) High nano- CaCO_3 content in cement mortar contributes to increased strength readings. The presence of nano- CaCO_3 helps reduce the variations in result values associated with higher DPF content.
- 5) According to the VIKOR technique, the mixtures containing 4% and 5% DPF, along with 3% and 4% nano- CaCO_3 , are the optimal choices.

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