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Research Article

Effect of Using Ozone Modified Waste Hempfiber on Some Fresh and Hardened State Properties of Cementitious Systems

Yagmur Basar^{1*}, Aliyea karsuozenc², Ali MardanI³ and Semiha EreN⁴

^{1,3}Bursa Uludag University, Faculty of Engineering, Department of Civil Engineering, Turkey

Corresponding Author:

Yagmur Basar. Bursa Uludag University, Faculty of Engineering, Department of Civil Engineering, Turkey.

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Abstract

It is known that various fibers are used to improve the brittle behavior of cementitious systems. In the production of artificial fibers used for this purpose, greenhouse gas emissions, which have become one of the biggest ecological problems of our day, occur in addition to economic reasons. This situation has caused the use of natural fibers in cementitious composites to become widespread. In addition, the reuse of waste materials in the construction sector has gained importance due to the increase in greenhouse gas emissions. In this context, the use of hemp waste fibers, which have an important potential in the development of environmentally friendly building materials, has become widespread in cementitious systems.

Since the low surface roughness and hydrophobic structures of natural fibers negatively affect the mechanical performance and durability of mortars, the fibers need to be modified with chemical and physical processes. In this study, the effect of the use of waste hemp fibers modified with ozone gas on the performance properties of cement-based mortars was investigated. Hemp fibers with a length of 6 mm were surface modified with ozone gas for two different periods (45 and 90 minutes) and were replaced with 0.5% aggregate by volume. In all mixtures, polycarboxylate-ether based high performance water reducing admixture was used by keeping the water/cement ratio 0.52 and the flow value 180 ± 20 mm constant. The flow, pressure and flexural strength of the mixtures were examined comparatively with the control mixture. It was observed that the flow performance of the mixtures was negatively affected by the use of fiber.

It was determined that the need for the admixture increased in mortar mixtures containing hemp. The water reducing admixture need of the mixtures was not affected by the fiber surface modification. With the addition of fiber, a significant decrease was observed in both the pressure and flexural strength of the mixtures. It was observed that the 45-minute ozone surface modification process applied to eliminate mechanical strength losses increased the pressure and flexural strength of the mixtures by strengthening the fiber-matrix interface.

Key Words: Cementitious Systems, Waste Hemp Fiber, Surface Modification with Ozone, FlowPerformance, Compressive, Flexural Strength

Introduction

Cement-based materials have disadvantages such as low tensile strength, tendency to crack and shrinkage, and the increase in brittle behavior as the strength increases can lead to significant problems in terms of structural integrity; therefore, fiber reinforcement stands out as an effective solution in eliminating these problems [1,2]. Synthetic fibers are widely used to provide such mechanical improvements, but they create environmental burdens and carry various health risks during production processes and usage stages [3,4]. This situation has made the use of natural fibers in building materials important as sustainable

and environmentally friendly alternatives.

In this context, hemp fibers (Cannabis sativa L.) are among the prominent natural reinforcement materials thanks to their high specific strength, biodegradability and sustainability [5-8]. The inability of natural fibers to provide sufficient adhesion with cement matrices may limit the expected performance increase [9]. Surface modification methods applied for the purpose of improving the fiber-matrix interface improve the interfacial interactions by changing the surface properties of the fibers

^{2,4}BursaUludag University, Faculty of Engineering, Department of Textile Engineering, Turkey

[10,11]. In this study, the effect of hemp fiber addition, which was surface modified with ozone gas, on the mechanical properties of mortar mixtures was investigated.

Ozone has been reported by various researchers to be a strong oxidant with an oxidation power of 2.07 eV [12,13]. It contributes to the formation of hydrophilic groups on the fiber surface and thus to the increase of adhesion to the cement matrix [14,15]. As a result of this process, significant improvements were observed in both flexural and compressive strengths,

demonstrating the positive effect of ozone application on the mechanical performance of cement composites.

Materials and Methods Materials

In this study, CEM I 42.5R Portland cement was used in accordance with EN 197-1 standard. The chemical composition and physical and mechanical properties of the cement supplied by the manufacturer are presented in Table 1.

Oxides	(%)
SiO ₂	18
Al ₂ O ₃	4.75
Fe ₂ O ₃	3.58
CaO	63
MgO	1.4
Na ₂ O+0,658K ₂ O	0.7
Specificgravity	3.06
SpecificSurfaceArea(cm ² /g)	3441

Table 1. Some properties of Portland Cement

CompressiveStrength (MPa)	7-Day	42.8
	28-Day	51.8
Setting Time(min)	Initial	170
	Final	240
SO ₃	3.11	

Table 2: Some properties of high-range water-reducing admixtures

In all fiber mixtures, 6 mm length hemp fiber was used in a constant ratio. Some properties of the hemp fiber used are shown in Table 3.

Density (g/cm3)	Lenght (mm)	Tensile Strenght (MPa)	Modulus of Elasticity (MPa)
1,5	6,0	500	12,7

Table 3: Some physical and mechanical properties of hemp fiber

Preparation of Mixture

In order to investigate the effect of surface modification process on cementitious systems, hemp fibers were subjected to ozone modification process. Thus, in addition to the control mixture, 3 different mortar mixtures were prepared in addition to 0.5% untreated hemp fiber and modified hemp fiber control mixture. In all mixtures, water/cement ratio was kept constant as 0.52 and flow value as 180 ± 20 mm. Water reducing admixture dosage was optimized for each 45 minutes of ozone surface modified properties of hemp fiber

mixture to provide the desired flow

value. Mortar mixture calculations were made for 1 m3 volume. The amounts of materials used in the production of mortar mixtures are presented in Table 4. The naming of fibrous mixtures was made according to fiber type and ozone duration. For example, the mixture in which untreated hemp fiber was used was shown as HF, while the mixture in which fiber was used was named as O45-HF.

Mixture	Cement	Water	Aggegate	PCE*	Fiber	Flow
Control	450	249,4	1534,0	0,6	-	188
HF	450	249,3	1522,8	2,1	7,4	185
O45-HF	450	249,3	1522,8	2,3	7,4	165
O90-HF	450	249,3	1522,8	2,0	7,4	185

Table 4: The amount of material used in the production of the mortar mixture (kg/m₂) and the flow values (mm)

Cement by Weight

In the scope of the study, river sand aggregate with a 0-4 mm sieve range, a specific gravity of 2.55 and a water absorption capacity of 2.34% according to the TS EN 1097-6 standard was used. In the mortar mixtures, a single type of polycarboxylate-ether based high-range water reducer was used in order to reach the target flow value of 180±20 mm. Some of the properties of the additive provided by the manufacturer are summarized in Table 2.

Method

The flow values of mortar mixtures were determined in accordance with ASTM: C1437 standard. ASTM C1437 standard test method is used to determine the flowability or flow diameter of self-compacting concrete. In general, the test

involves placing a sample of the material in a stacking cone, then lifting it and allowing it to flow on a flat surface. The resulting flow diameter is measured and used as an indicator of the flowability of the material. Compressive strength and flexural strength of mortar mixtures were determined in accordance with EN 196-1 standard.

EN 196-1 standard test method is used to determine the flexural and compressive strength of cement mortars. The test involves placing the mortar prepared in standard proportions into molds and waiting under certain curing conditions. First, flexural strength is applied to the samples at specified ages, and then compressive strength is applied on the separated pieces.

Fiber Surface Modification

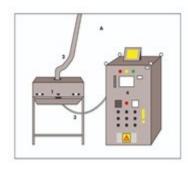




Figure 1: Ozone generator and ozonation device [15]

Experimental Results and Discussion

Flow Performance

The amount of water-reducing admixture required to achieve the target flow value of the mixtures is given in Figure 2.

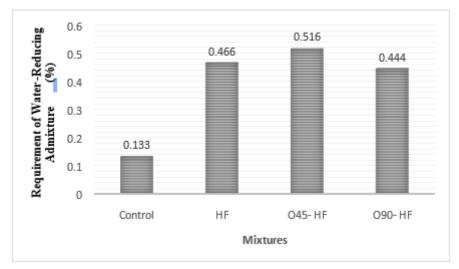


Figure 2: Water reducing admixture requirements of mixtures

The ozonization system used in the surface modification of hemp fibers is presented in Figure 1. Ozone gas was produced in a 25 g/h capacity Opal PRO DO25 O2 generator according to the corona discharge method. In this study, hemp fibers were ozonated for 45 and 90 minutes using an ozone concentration of 15 L/min. The processes were carried out separately for both periods. The ozone surface modification system of hemp fibers is presented in detail in Figure 1. In Figure 1, A, the lid of the ozonization system is shown closed, while B. shows the lid of the ozonization system open. The numbers on the visual in Figure 1 indicate, respectively, 1. ozonization system, 2. ozone

discharge pipe, 3. ozone feeding pipe, 4. ozone generator, 5. templates, and 6. ozone discharge fan. The hemp fibers were placed between the templates indicated with number 5 in Figure 1 and the ozonization process was carried out in a closed manner (A).

According to the data presented in Figure 5, it was determined that the addition of hemp fiber significantly increased the amount of water-reducing additive required for mortar mixtures to reach the target flow value, regardless of the ozone treatment. With the addition of hemp fiber, a 3-3.8 times higher water-reducing

additive requirement was observed compared to the control mixture. The porous structure and high cellulose content of hemp fiber increase the sensitivity of hemp fiber to moisture, which can negatively affect its performance in various applications. [16]. Cellulose is a hydrophilic (water-attracting) material, therefore, it absorbs water in the mixture and causes the amount of free water to decrease. [17,18]. This situation negatively affects the workability of the mixture and leads to a decrease in flow values. It was determined that the additive requirement in fibrous mixtures did not cause a significant change in the additive requirement, regardless of the modification process time. The water-reducing additive requirement of the mixtures was not affected by the fiber surface modification.

Compressive and Flexural Strength

The 7-day compressive and flexural strength results of the mortar mixtures are shown in Figure 3. Regardless of the modification process, it was determined that the compressive strength values of the fibrous mixtures were generally lower than the control

mixture. This is thought to be due to the hemp fibers not being mixed homogeneously, thus forming voids by clumping. Similarly, in a study conducted by Mardani Aghabaglou it was emphasized that the strength was negatively affected due to the risk of clumping in the matrix due to the presence of excess fibers in the system and the increase in the void volume [19]. However, it is thought that the hemp fiber's high water absorption capacity makes the mixture cohesive and the fibers entrain air during mixing.

The compressive strength of the HF mixture was determined as 12.43 MPa. The compressive strength of the O45-HF mixture, which was subjected to 45 minutes of ozonization, increased to 13.92 MPa and an improvement of 12.0% was achieved. This situation is thought to be due to the improved adhesion between the fiber-matrix that increased with the modification. As a result of the O90-HF mortar mixture, which was subjected to 90 minutes of ozonization, the compressive strength decreased to 9.61 MPa and a decrease of approximately 31.0% was experienced.

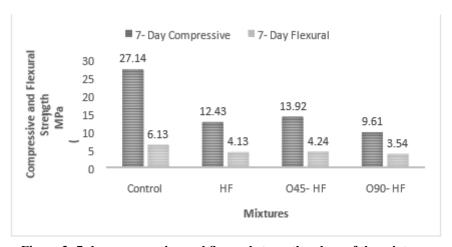


Figure 3: 7-day compressive and flexural strength values of the mixtures

It was observed that the flexural strength decreased with the addition of fiber to the mixture, regardless of the modification process. It is thought that this situation may be due to the fact that the fiber is not mixed homogeneously due to the excess in the matrix, thus increasing the void volume. [20]. The flexural strength of the HF mixture was measured as 4.13 MPa. In the sample that was subjected to 45 minutes of ozonization, this value increased to 4.24 MPa and an increase of approximately 2.8% was observed. The fibers prevented crack propagation with the bridging effect and reduced micro-crack formation, resulting in an improvement in the flexural strength of the mixtures [21,22]. In the study conducted by Şahin et al it was reported that an increase in the flexural strength of ozonated fiber-reinforced mixtures was observed [15]. In the sample containing hemp fiber that was ozonated for 90 minutes, the flexural strength decreased to 3.54 MPa, showing a decrease of approximately 16.7% compared to the untreated state. There was a decrease in the flexural and compressive strength values in the samples that were ozonated for 90 minutes compared to the result of 45 minutes of ozonization.

It is thought that this situation is due to the damage to the structural integrity of the fibers with the increase in the process time [23]. The findings obtained revealed that the 45-minute ozonization period positively affected the mechanical properties and provided

improvements in both bending and compressive strength. It shows that the 45-minute ozone surface modification process applied increased the compressive and bending strength of the mixtures by strengthening the fiber-matrix interface. However, when the ozonization time was increased to 90 minutes, it was determined that the structural integrity of the fibers was damaged and significant decreases in mechanical properties occurred. In line with these data, increasing the performance of hemp fibers through surface modification is considered an important method in the development of environmentally friendly, durable and economical cement-based composites.

Conclusion

The materials used in the study and the findings obtained as a result of the experiments performed are summarized below.

It has been observed that the demand for water-reducing additives to reach the target workability value increases with the addition of fibers to the mixtures. It has been determined that the need for additives in fiber mixtures does not cause a significant change in the need for additives, regardless of the modification process time. It has been observed that the effect of surface modification with ozone on the pressure and flexural strength of mortar mixtures varies depending on the applied process time. A 12% improvement was observed in the compressive strength

of mortar mixtures containing hemp fibers that underwent a 45-minute surface modification process with ozone compared to the untreated mixture. A 31% decrease was observed in the compressive strength of mortar mixtures containing hemp fibers that underwent a 90-minute surface modification process. A 31.0% decrease was observed in compressive strength as a result of 90 minutes of ozone application. In the sample that underwent 90 minutes of ozone treatment, the flexural strength decreased by approximately 16.7% compared to the untreated condition.

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