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# Flexural stress enhancement of concrete by incorporation of algal cellulose nanofibers





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# HIGHLIGHTS

• The cellulose nonofiber was succesfully derived from Cladophora sp green algae which is abundant in nature as a waste form.

• Flexural stresses of the concrete samples were measured by the three-point bending test.

• The concrete samples' flexural stress increased 2.7 times by adding algal cellulose nanofiber in concrete mortar.

# ARTICLE INFO

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# ABSTRACT

Using nanofibers as a reinforcement material in the concrete and its positive effect on the concrete strength is one of the most popular topics in recent years. In the present study it was demonstrated that the nanofibers derived from waste algae (Cladophora sp) increased 2.7 times the flexural stress of the concrete. Firstly, the cellulose nanofibers were derived from Cladophora sp algae which stand in the nature as a waste and can be collected in tones. The obtained cellulose nanofiber and the commercial cellulose were characterized by Fourier Transform Infrared (FT-IR) Spectroscopy, Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), X-ray diffractometry (XRD) and Scanning Electron Microscopy (SEM) methods. Then, various amounts of the cellulose nanofiber and the commercial cellulose were added in the cement paste. Before the flexural stress tests, the concrete samples were left to dry for one week. Flexural stresses of the concrete samples were measured by the three-point bending test. The flexural stress of the control concrete sample (with no nanofibers) was measured as 2.21 MPa. The flexural stress of the concrete sample added 1.0 g cellulose nanofiber (derived from algae) were measured 5.96 MPa. On the other hand, it was determined that the commercial cellulose fibers decreased the flexural stress of the concrete samples. The used *Cladophora* sp algae in this study has a worldwide potential of extreme algal blooming in the aquatic ecosystems and the algal blooming of Cladophora sp causes to eutrophication of water sources. In this study it was proven that removing of algal mats from nature and using them as a reinforced material in concrete can be wisely solution instead of the synthetic cement paste additives.

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# 1. Introduction

The usage of several plants' straw as a reinforcement material in construction supplies dates back to the first centuries. As is known, straw contains between 30 and 40% cellulose [1]. So as the cellulose as a reinforcement material in straw is known to play an important role. Based on this, obtaining pure cellulose nanofiber from plants and effects of cellulose nanofibers' on concrete strength by adding them in cement paste have been wondered.

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http://dx.doi.org/10.1016/j.conbuildmat.2017.05.104 0950-0618/© 2017 Elsevier Ltd. All rights reserved. Because of nanofibers' highest length/diameter ratio (aspect ratio), nanofibers show an improved elastic modulus and strength at break [2]. First in 2009, it is observed that reinforced concrete with cellulose nanofibers, significantly improved the strength of the concrete [3]. Then, highly improved concrete strength was revealed by the reinforcement of concrete with cellulose nanofibers obtained from sisal, cotton [4] and wood [5]. Similarly, in this study; the effects of cellulose nanofibers obtained from fibrous algae on concrete flexural stress have been researched.

Pressure, tensile and bending tests are mechanical tests applied on hard concrete. Bending test is the most widely used method for measuring the mechanical strength of fiber containing concrete because, when the concrete receives the load, stretching without breaking and to prevent breakage is essential for concrete systems [6]. In this study it is figured that reinforcing the concrete samples with the nanofibers increases the flexibility. Therefore, the bending mechanical tests were made to calculate the flexural stress of the concrete samples.

*Cladophora* sp is a filamentous algae, which covers the coastal line of lakes and seas by the algal blooming, especially in hot climates. Similarly, they cover the entire water surface in case of decreasing the water level in rivers. The species of this genus have the proliferative potential in a short time on the surface of water, and they have a widely distribution throughout the world. In case of overbreeding, if they cannot be removed from water they can cause eutrophication [7,8]. The reasons to select *Cladophora* sp as a nanofiber source are; its prevalence around worldwide and the collection of this waste algae can be beneficial for the environment.

In an earlier study, the cellulose obtained from *Cladophora* sp usage as a construction material in polyurethane foams, fish food, filter, drug carrier, rheology enhancers and conductive composite material were researched [9–13]. In this study nanofibers were derived from *Cladophora* sp which has a waste form in a river and it was characterized by FT-IR, TGA, DSC, XRD and SEM, comparatively with commercial cellulose. Then the effect of algal cellulose and commercial cellulose reinforcement on flexural stress of the concrete samples was investigated.

#### 2. Experimental methods

### 2.1. Materials

Cellulose nanofibers used in this study were obtained from *Cladophora* sp which was collected from Mamasın Dam Lake (Aksaray, Turkey) in November 2013. And commercial cellulose nanofibers were supplied from cotton (Sigma-Aldrich, CAS: 9004–34-6).

#### 2.2. Bleaching

20.0 g powdered *Cladophora* sp sample was refluxed 20 min with 200 mL sodium hypochlorite (NaOCl) 4.0% v/v (10–14% w/w) solution at 150 °C. After this treatment, a white algal sample was obtained.

#### 2.3. Deproteinization

After the bleaching process the samples were refluxed with 2 M sodium hydroxide, (NaOH, 4 mol/L) at 150  $^\circ$ C for 2 h. Then the

samples were rinsed with distilled water until reaching the neutral pH. This process was performed to remove proteins from the structure.

#### 2.4. Removing of oil and pigments

After the deproteinization, the samples were refluxed 20 min with chloroform (CHCl<sub>3</sub>, 1.492 g/mL), methanol (CH<sub>3</sub>OH, anhydrous, 99.8%) and distilled water mixture (4:2:1 by volume). Then the samples were rinsed with distilled water until pH 7.0. After this treatment, the oil and pigments were removed from the cellulosic material.

# 2.5. Demineralization

Finally, the samples were refluxed with 2 M 100 mL hydrochloric acid, HCl  $(1.1 \text{ g/cm}^3)$  solution at 100 °C for 10 h. After this treatment, the minerals were removed and algal cellulose was obtained. The wet samples were left to dry at 40 °C for a week. Then, white colored cellulose nanofibers were obtained.

# 2.6. Preparing fiber reinforced concrete (FRC) samples

Concrete mortars were prepared with 80.0 g fine sand (diameters < 2.0 mm), 20.0 Ml distilled water, 20.0 g Portland Cement (Elazığ, Altınova Cement Factory Inc. PC 40.0) and various amounts (0.1 g, 0.25 g, 0.5 g, 0.75 g, 1.00 g) cellulose nanofibers. Also concrete samples without fiber reinforcement were prepared for comparison purposes. These ratios were adapted from Mohamed, Ghorbel [14]. The wet concrete mortars were laid in plastic mold ( $38 \times 13 \times 120$  mm) and left to dry 7 days before the flexural test

#### 2.7. The three point bending flexural test

The flexural stress value of the materials was provided by the three-point bending flexural test (Fig. 1). Flexural stress is a mechanical parameter for brittle material, defined as a material's ability to resist deformation under load. Fiber Reinforced Concretes (FRC) have higher flexural stress because they become more flexible by fibers.

The concrete samples with  $38 \times 13 \times 120$  mm dimensions were located to the Baehr Universal Test Machine's supports. The velocity of the force which was applied to the samples' midpoint is 100 mm/min. The highest forces before the breaking were used at the flexural stress equation:  $f_{ef} = \frac{3FL}{2d_1d_2^2}$  [15]



Fig. 1. Three point bending flexural test.

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