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Effects of Treated Waste Wood Fibers Incorporation on Rheological Properties of the Construction and Demolition Waste-based Geopolymer Mortar

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Abstract

Nowadays, the enormous amount of generation of construction and demolition waste (CDW) is a major problem, considering its detrimental effects on the environment. In this content, this study focused on the valorization of waste-wood fibers in CDW-based geopolymer mortar. Chipped waste-wood fibers (WWFs) (~2.00 mm) were chemically treated by immersing them into alkaline solutions (2.5 M NaOH (Sodium hydroxide) solution) for two hours to reduce inorganic impurities attached to the surface of fibers, in favor of increased compatibility with the geopolymer matrix. Then, treated WWFs were incorporated into the CDW-based mortar mixture activated with NaOH and $Ca(OH)_2$ (Calcium hydroxide). To assess the effects of fiber inclusion on the rheological properties of CDW-based geopolymer mortar mixture, empirical test methods of flow table, vane shear test, and buildability tests were conducted and rheological tests with an advanced rheometer device were performed by different implementation protocols of flow curve and three interval thixotropy. The results showed that the incorporation of chemically treated WWFs yielded an increment in static and dynamic yield stresses and viscosity of CDW-based geopolymer mortars. Besides that, the inclusion of wood fibers caused increments in the thixotropy performance of mixtures, providing better viscosity recovery performance after the agitation. It is believed that the findings of this study will contribute to the current literature by proposing a different way of producing novel CDW-based geopolymers and valorization of WWFs in construction materials.

Keywords

Construction and demolition waste, Geopolymer, Waste-wood fiber, Upcycling, Rheological properties

Introduction

As cities transform and develop, many buildings at the end of their service life or in a dangerous condition for the environment were demolished and it is expected that this demolition process will continue continuously due to the development activities of the countries [1-3]. CDWs generated from these demolition activities are mostly stored in clean landfilling areas. This disposal process of CDWs requires very large areas and is inefficient in terms of health, economy, and environment [4], because depositing waste in clean lands can lead to contamination, posing significant risks to human health and the environment. This contamination can have severe and detrimental consequences. The success of the countries in the world in properly handling CDWs varies considerably and is mostly limited to old-fashioned methods such as direct crushing and road base/subbase filling. This inefficiency in CDW valorization has triggered the development of innovative and effective ways to use CDWs, especially in developing countries. When considering that CDW production is a global problem and CDW can be found almost anywhere in the world [5, 6], an effective way to address CDW disposal is an urgent need. The usage of CDW-based materials in the production of geopolymer systems can be a proper method for handling CDWs [5, 6]. For more effective and innovative waste management/control and high-grade utilization, CDW-based components (e.g., concrete, tiles, ceramics, bricks, and glass) can be used as aluminosilicate precursors together with different-sized recycled aggregates in the production of geopolymer systems. This kind of utilization can make these waste materials valuable secondary raw materials for the construction industry/applications. This upcycling/recycling process is beneficial for effectively minimizing the number of CDW-based materials that end up in clean landfills and reducing the amount of concrete production, as well as cement and virgin aggregate production [5-9].

Despite the efforts on the valorization of CDW-based materials, 75% of waste wood originated from construction and demolition activities, which corresponds to 7% of the whole CDWs, cannot be incorporated in the value-added chain and is kept in landfills, while the remainder is used as secondary material for use as fuel, compost, or other materials [10]. Wood is a natural fiber structural composite that has been used by people for thousands of years as a building material and to generate heat energy. The wood industry sector practically uses raw materials as a whole. Therefore, wood waste, a residue from construction wood that is unsuitable for construction activities, is either mechanically ground to produce fibers or chips or chemically treated to prepare cellulose. Studies in the literature have shown that this waste can be used as coatings, chips, or panels in different areas after proper pretreatment processes and improves mechanical properties when incorporated into the construction materials (e.g., concrete and geopolymer) as a fiber [11-15]. Recent studies have focused on the improvement of basic engineering properties (flexural tensile, shear, ductility, energy absorption capacity, etc.) of cement-based composites via the incorporation of wood fiber in favor of obtaining superior mechanical performance [15]. In addition to this, the incorporation of wood fibers in geopolymer systems is currently being tried. Ye et al. [16] conducted a study on the interfacial bonding properties of wood fiber-containing geopolymer composites. They found that increasing the roughness of the wood surface improved the interfacial bonding between wood and geopolymer matrix. The rough surface of the wood fiber facilitated a strong bond with the surrounding geopolymer paste, resulting in higher compressive strength of the composite. Su et al. [17] examined the effect of lignin fiber on the strength and linear shrinkage of geopolymer. The results showed that increasing the amount of lignin fiber in the geopolymer composite led to an improvement in strength and a reduction in linear shrinkage. This suggests that the incorporation of lignin fiber can enhance the mechanical properties and dimensional stability of geopolymer materials. In the study of Ye et al. [18], the influence of lignin, cellulose, and hemicellulose on geopolymer-based composites was investigated. A low content (5 wt.%) of these components was found to enhance the flexural and compressive strength of geopolymer. However, higher amounts of lignin and hemicellulose resulted in porous structures, lower density, and brittle fractures, leading to reduced strength in the composites. In a study conducted by Lin et al. [19], it was found that incorporating wood fiber into geopolymer system has positive effects on reducing brittleness and increasing fracture toughness. This improvement was attributed to the strong bonding between the reaction products and wood fiber. However, the addition of wood fiber had negative consequences for the setting and hardening behavior, reaction degree, and compressive strength of the geopolymer. All the results of these studies showed that an improvement in some engineering properties of geopolymer materials can be obtained by the incorporation of wood fibers [including waste (WWF)]. To this motivation, the incorporation of waste wood as fiber into CDW-based geopolymer systems can be a move that increases efficiency in favor of waste recycling.

Although the performance of cellulosic fibers in various composites used in construction activities is determined by the characteristics of the fiber, the surfaces on which the fibers are exposed to various effects influence the performance of the composites [20]. CDW-based wood fibers can have lower tensile capacities compared to the fibers obtained from newly produced wood because of the various environmental effects (moisture, abrasion, etc.) to which their surfaces are exposed. Therefore, there is a further need to improve the surface properties of CDW-based fibers. Various physical (cold plasma treatment, gamma ray method, electrical charging, corona treatments, and nanomaterial impregnation) and chemical methods (peroxide, permanganate, binding agents, etc.) are used to improve the properties of natural fibers such as wood fiber [21]. Some of these methods require various additional intermediate processes and hence they result in high costs. In order to use the wood fiber more efficiently in the composite systems, the impregnation method, which is one of the more practical and less costly methods, can be adopted. In this method, impregnation of matrices (thermoplastic, polymer, and alkaline based) in which natural fibers are used with dilute solutions significantly improves the mechanical properties of the fibers [22]. In one of literature studies, Barman et al. [23] examined the impact of different concentrations of NaOH (ranging from 2.5% to 20%, with increments of 2.5%) on pine wood fibers. The findings indicated that a NaOH concentration of 10% resulted in a significant removal of amorphous lignin, while concentrations above 10% led to a decrease in cellulose content. The researchers also noted that increasing the NaOH concentration beyond 10% led to the deterioration of the crystal structure of cellulose, alongside the elimination of lignin and hemicelluloses. Raia et al. [24] investigated the effects of NaOH treatment (at the concentrations 5, 10, 15, 20, and 30%) on the wood-derived cellulose fibers. The results indicated that higher concentrations of NaOH resulted in increased thermal stability and improved surface characteristics of the fibers. The study demonstrated that the cellulose fibers' properties could be modified effectively by NaOH treatment. The condition involving a 5% NaOH concentration and a 2-hour alkalization duration exhibited the most desirable results, making the treated fibers suitable for utilization as fillers in plant-based composites. In the study conducted by Agarwal et al. [25] sawdust was treated with NaOH to enhance its suitability as a reinforcing filler in composites. The results of the study demonstrated that treating the sawdust with NaOH led to a significant improvement in the mechanical properties of the resulting composites compared to those made with untreated sawdust. Specifically, the flexural strength of a composite consisting of 10% treated sawdust as a hybrid filler surpassed that of an epoxy resin composite reinforced with glass fibers. This finding indicates that the NaOH treatment effectively enhanced the strength and structural integrity of the composite material. All these results proved that alkali treatment is beneficial to improve in some engineering properties of natural fibers. From this point of view, it is more important to improve the surface properties of CDW-based fibers as mentioned before, which have been exposed to different conditions during their service life and have more residues/dust/dirt on the surface compared to virgin natural fibers, in terms of efficient use of them in composite production. In addition, with the aim of popularizing the high-grade valorization, it is very crucial to perform studies to increase the efficiently use of CDW-based fibers with improved surface properties through alkalinization in waste-based geopolymer systems (especially in CDW-based geopolymer systems). It is clear that the rheological and mechanical properties of geopolymer systems are affected with the incorporation of CDW-based fibers, and studies on this also need to be done.

Within the scope of this study, the characteristic mechanical properties of WWFs were tried to be strengthened via the impregnation method by using alkaline activators to be prepared in dilute form. In favor of facilitating the combination of cellulose and alkaline activators and providing a more advanced interface, the alkaline activator solution was tried to be used to eliminate hemicellulose, lignin, pectin, and various wood oils on the WWFs. After that, CDW-based geopolymers were incorporated with treated CDW-based WWFs. Different CDW-based materials hollow brick (HB), red clay brick (RCB), roof tile (RT), glass (G), and concrete rubble (C) were used as aluminosilicate precursors. For the alkaline activation of geopolymers, NaOH, and $Ca(OH)_2$ were used. At the end of the study, the effect of the treated CDW-based WWFs on the rheological properties of geopolymer systems was evaluated by performing a flow table test, vane shear test, rheometer tests following flow curve and three interval thixotropy test (3ITT) protocols.

Experimentation

Material and mixture proportion

In this study, RCB, HB, RT, G, and C were used as CDW-based aluminosilicate precursors which were collected in a form of rough fragments from the demolition sites. To obtain powder form to be able to use them as a precursor, the rough fragments were crushed first in the jaw crusher to reduce size. Then, the milling process was applied to the crushed materials for 1-hour to obtain the ultimate powder form. After the milling process, while the maximum particle size of RCB, HB, and RT was recorded as approximately 50 μm, the maximum particle size of G and C was about 120 μm because of difference in grindability characteristic of precursors. Wood fibers are nanoscale materials. The physical appearance of the CDWs was represented in figure 1. Oxide composition and specific gravity of the CDW-based precursors were given in table 1. According to X-ray fluorescence results, major oxides in RCB, HB, and RT were SiO_2, Al_2O_3, and Fe_2O_3 while the main oxides of C and G were recorded as SiO_2 and CaO.

Fine recycled concrete aggregate (RCA) was included in the mixture design to prepare CDW-based mortar mixtures. Collected concrete rubble was crushed in a jaw crusher to reduce the size. Then, crushed RCAs were sieved to obtain RCA having a maximum particle size of 2 mm. The physical appearance of the RCA was represented in figure 1.

Table 1: Oxide compositions and specific gravity of CDW-based materials.

Oxides, %	HB	RCB	RT	G	C
SiO_2	39.7	41.7	42.6	66.5	31.6
Al_2O_3	13.8	17.3	15.0	0.9	4.8
Fe_2O_3	11.8	11.3	11.6	0.3	3.5
CaO	11.6	7.7	10.7	10.0	31.3
Na_2O	1.5	1.2	1.6	13.6	0.5
MgO	6.5	6.5	6.3	3.9	5.1
SO_3	3.4	1.4	0.7	0.2	0.9
K_2O	1.6	2.7	1.6	0.2	0.7
TiO_2	1.7	1.6	1.8	0.1	0.2
P_2O_5	0.3	0.3	0.3	0.0	0.1
Cr_2O_3	0.1	0.1	0.1	0.0	0.1
Mn_2O_3	0.2	0.2	0.2	0.0	0.1
Loss on ignition	7.8	8.0	7.5	4.3	21.1
Specific gravity	2.9	2.8	2.9	2.5	2.7


(a) (b) (c)

Figure 1: The physical appearance of the CDW-based materials: **(a)** RCB, HB, RT, G and C from left to right, **(b)** RCA, and **(c)** WWFs treated with 2.5 M NaOH for 2 hours.

To activate CDW-based geopolymer mixtures, NaOH and $Ca(OH)_2$ were used in this study. NaOH was in flake form and has a purity level of 98%. $Ca(OH)_2$ was in powder form and has a purity level of 87%.

To assess the effects of the WWFs on the fresh properties of the CDW-based geopolymer system, waste woods were first collected from the demolition sites. Then waste wood was chipped and sieved to obtain fibers having a maximum size of 2.00 mm. The average length, width, and depth of the WWFs were measured as 12.45 ± 3.53 mm, 1.3 ± 0.23 mm, and 0.5 ± 0.18 mm, respectively. The aspect ratio was determined as 10 for the obtained WWFs. As the waste wood contains some impurities because of both processes applied during production (painting, covering, etc.) and its own nature (hemicellulose, lignin, wax oil, etc.), WWFs were treated with 2.5 M NaOH solution for 2 hours to eliminate impurities and enhance fiber properties (Figure 1) [26]. So, WWFs treated with 2.5 M NaOH for 2 hours were used in this study to investigate the effects of WWFs on the fresh properties of CDW-based geopolymer.

Considering the chemical content (Si, Al, Ca, etc.) and knowledge from the previous studies conducted [7, 27], mixtures were designed. Approximately 80% of the dry base mixture consisted of clay-based waste materials (HB, RCB, and RT), while both C and G were utilized in equal proportions, comprising 10 + 10% of the total weight of the mixture. It is noteworthy that the oxide compositions and particle size distributions of HB, RCB, and RT were similar, resulting in their equal usage, which accounted for 26.67% of the total weight of the base mixture (as shown in table 2). C and G were added in limited quantities to enhance the composition of the base mixture in terms of CaO and SiO_2, without significantly altering the balance of Al_2O_3. The detailed mixture proportion of both CDW-based mortar and CDW-based composite containing 2% (in volume) treated WWFs were shown in table 2. While the water-to-binder ratio (by weight) was 0.33, the aggregate-to-binder ratio (by weight) was 0.35. However, for the test methods conducted by rheometer, water-to-binder ratio (by weight) was decided as 0.39 to provide more precise results considering the measurement capacity of the rheometer. While NaOH molar concentration of 10.0 was used, $Ca(OH)_2$ was included with the utilization ratio of 4.0% (by weight of total precursor).

Test methods

Flow table test

The flow table test was conducted by following the ASTM C1437 standard. To demonstrate results more understandable, the results were converted to the flowability index (Γ) by applying the equation given below.

$$\Gamma = \frac{d_1 d_2 - d_0^2}{d_0^2}$$

Where, d_0 is the inner diameter of the mold (100 mm), d_1 is the maximum spreading diameter and d_2 is the spreading diameter perpendicular to d_1. The higher the flowability index, the better the workability performance. The flow table test was repeated for time intervals of 30, 60, and 120 min, in addition to the initial fresh state.

Buildability test

The buildability performance of the mixtures, which is also called the shape-retention performance, was measured by following the test standard proposed by Nematollahi et al. [28]. First, the materials were placed on the mini-slump cone and kept in the mold for 1 minute. Then, the mold was removed, and the static load of 600 g was placed on the top surface of the cone-shaped materials for 1 minute. In the end, the ultimate height of the mixture was measured. The higher the ultimate height (higher viscosity or yield stress), the better the buildability performance.

Vane shear test

Vane shear test was conducted to measure the fresh properties of CDW-based geopolymer mixtures. To perform the test, a pocket-type vane shear tool was used. During the test, the vane apparatus measures the resistance of the materials to the shear force applied. The test was repeated for time intervals of 30, 60, and 120 min, in addition to the initial fresh state. The higher the vane shear stress, the higher the viscosity.

Flow curve test protocol (rheometer)

Flow curve analysis is a protocol that is applied by using a rheometer. The test provides to obtain the static and dynamic yield stress of the mixtures. This test was conducted in this study by following three steps. Pre-shear having a test protocol of linearly increasing shear rate from 0.1 s^{-1} to 100 s^{-1} was applied first to provide homogeneity in mixtures, elimination of air bubbles/pores, and similar shear history for each mixture. After that, 30 seconds of resting time followed. Then, a shear load was applied with a linearly increasing shear rate from 0.1 s^{-1} to 100 s^{-1}. After that stage, 30 seconds of resting time followed. Then, a shear load was applied with a linearly decreasing shear rate from 100 s^{-1} to 0.1 s^{-1}. The static yield stress was determined from the first peak of measured data in shear stress vs shear rate of the pre-shear test protocol, as the material was in the undisturbed state in the initial phase [29-31]. The dynamic yield stress was determined by applying the Bingham model to the shear stress vs shear rate results of the last protocol as the materials become more homogenous as a result of the longer applied shearing force.

Table 2: Mixture proportions of CDW-based systems.

Mixture code	CDW-based precursor (g) (1000g)					RCA (g)	WWF (g)	Alkaline activator	
	RCB	HB	RT	G	C			NaOH (g)	$Ca(OH)_2$ (g)
CDW	266.67	266.67	266.67	100	100	350	0	132	40
CDW-F	266.67	266.67	266.67	100	100	350	13.4	132	40

3ITT protocol (rheometer)

3ITT protocol was applied to the materials by using a rheometer. The test was conducted to measure the viscosity recovery performance of the mixtures which is also known as the thixotropy performance of the mixtures (Figure 2). The test was conducted by applying the shear rate described in the following steps: stage (i) shear rate of 0.1 s^{-1} for 60 seconds, stage (ii) shear rate of 10 s^{-1} for 10 seconds, and stage (iii) shear rate of 0.1 s^{-1} for 40 seconds. During the test, corresponding shear stress to the applied shear rate was recorded and the thixotropy performance of the mixture was calculated by comparing the viscosity value at the stage of (i) and (iii).

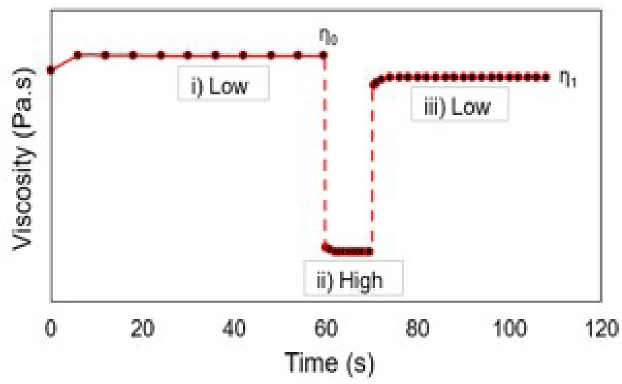

Figure 2: 3ITT protocol.

Results and Discussion

Table 3 shows the results obtained after performing the time-dependent flow table and vane shear tests on both the mixture without fiber and the fiber-incorporated mixture. It can be followed from this table that irrespective of open time, the flowability index values of geopolymer mixtures containing fibers were lower compared to those of fiber-incorporated geopolymer mixtures while their vane shear stress values were higher. The decrement in fluidity with the incorporation of the fiber into the mixtures can be attributed to the increase in the total amount of solids present in the system and the absorption of the water in the system by the incorporated fibers. For all geopolymer mixtures, most probably due to the ongoing geo-polymerization reactions, vane shear stress values increased with time while the flowability index values decreased. The amount of change in test results (both of flow table and vane shear test) for the fiber-containing mixture was lower compared to that for the mixture without fiber and this means that the time-dependent variations in the rheological properties of the mixtures were reduced with the fiber incorporation. The possible reasons for these results were discussed below.

Table 3: Flow table and vane shear test results.

Test parameters		Open time			
		0 min	**30 min**	**60 min**	**120 min**
Flowability Index (Γ)	CDW	0.756	0.729	0.638	0.440
	CDW-F	0.625	0.525	0.452	0.402
Vane Shear Stress (N/cm^2)	CDW	1.6	3.5	3.9	5.6
	CDW-F	4.3	5.2	5.8	7.1

Buildability, flow curve, and 3ITT test results were presented in table 4. According to the buildability performance results which are associated with the static yield stresses of the mixtures, the inclusion of the WWFs resulted in an increment in the buildability performance of the CDW-based geopolymer mixture by about 12%. Meanwhile, static yield stress results obtained from the flow curve test protocols showed that the inclusion of WWFs yielded an increment in the static yield stress of the mixture by approximately 119%. Flow curve test results showed that the incorporation of WWFs into the CDW-based geopolymer mixture also resulted in an increment for dynamic yield stress, in addition to the static yield stresses. This can be explained by the increase in the solid content of the matrix and the decrease in the pore water as a result of the high-water absorption of treated fibers (compared to other components) trapping/collecting the void water in the system. However, an increment in dynamic yield stress was calculated at approximately 87% which was quite lower compared to the static yield stress increment obtained from the flow curve test. This could be explained in a way that during the undisturbed stage, the WWFs may absorb free water in the matrix which could yield a more increment of static yield stress, yet after shear stress applying (squeezing), WWFs may leave the absorbed water which may lead to less increment in dynamic yield stress compared to the static yield stress. According to the 3ITT results, the inclusion of WWFs led to an increment in the recovery performance of the CDW-based geopolymer mixture by approximately 5%. The increase in thixotropy performance means that the material can get back closer to its initial steady-state viscosity after agitation. In this case, it has been demonstrated that the added fiber allowed the developed geopolymer material to recover its viscosity more after deforming. The possible reason for this situation could be that the wood has a rigid structure because when the shear force (deforming force) was removed, the fiber tended to stay stationary/rigid in the matrix which may provide fast viscosity recovery. Besides that, obtained results from the flow curve tests were also in coincide with the 3ITT. In the study of Qian and Kawashima [32], it was stated that the differences between dynamic and static yield stress are attributed to the thixotropy phenomena. Since WWFs inclusion caused more increment in static yield stresses and less increment in dynamic yield stress, the inclusion of WWFs may cause the increment in the thixotropy performance of the CDW-based geopolymer mixture. So, the obtained results regarding the thixotropy performance of the mixture from the flow curve and 3ITT were consistent with each other.

Table 4: Buildability performance and rheometer test results of CDW-based geopolymer mortar and WWFs-incorporated CDW-based geopolymer composites.

Test parameters		CDW	CDW-F
Buildability Performance (cm)		4.2	4.7
Flow Curve	Static Yield Stress (Pa)	2193.1	4798.8
	Dynamic Yield Stress (Pa)	832.0	1555.2
3ITT	Recovery Performance (%)	76.2	80.1

Conclusions

The following conclusions were drawn from the experimental studies performed within the scope of the current study:

- Irrespective of open time, lower flowability index values and higher vane shear stress values were obtained from geopolymer mixtures containing fibers compared to those of fiber-incorporated geopolymer mixtures.
- For all geopolymer mixtures, vane shear stress values increased with time while the flowability index values decreased and the amount of change in test results (both of flow table and vane shear test) for the fiber-containing mixture was lower compared to those for the mixture without fiber.
- Inclusion of WWFs yielded increment in both static and dynamic yield stresses of CDW-based geopolymer. Moreover, the incorporation of fibers resulted in increment of viscosity recovery performance of the mixture.

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Conflict of Interest

None.

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