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Compressive Strength of Geopolymer Mortar Reinforced with Rice Husk Fibers

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ABSTRACT

Geopolymer concrete has been proposed to minimize carbon dioxide emissions related to the cement production industry. This environmentally friendly material consists of industry waste materials activated chemically by an activation solution. In this study, the geopolymer mortar has been designed with 70% fly ash and 30% metakaolin. Hydroxide sodium in 14 molar concentrations blended with sodium silicate in a 2.5:1 ratio was used as the activation solution. Rice husk fibers were added as reinforcement in (1%, 1.5%, and 2%), and waste paper (paper pulp and paper ash) was added in (1%, 2%, and 5%) by volume of cementitious material. The geopolymer mortar samples underwent a curing process through exposure to a temperature of 60°C in an oven for 24 hours. The findings indicate that the samples reinforced with 2% rice husk fibers exhibited the most significant improvement in compressive strength, with 59% and 55% increases after 7 and 28 days of curing, respectively. In general, using waste paper and rice husk fibers significantly enhanced the compressive strength of geopolymer mortar.

Keywords: Geopolymer mortar, Fly ash, Metakaolin, Compressive strength, Rice husk fibers, Waste paper.

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مقاومة الانضغاط لمونة الجيوبوليمر المسلحة بألياف قشور الرز

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قسم الهندسة المدنية، كلية الهندسة، جامعة بغداد، بغداد، العراق

الخلاصة

تم اقتراح الخرسانة الجيوبوليمرية كوسيلة لتقليل انبعاثات ثاني أكسيد الكربون الناتجة من صناعة انتاج السمنت. تتكون هذه الخرسانة الصديقة للبيئة من مخلفات المصانع والتي يتم تفعيلها كيميائيا عن طريق محاليل مفعلة. في هذه الدراسة تم تصميم مونة الجيوبوليمر باستخدام 70% من رماد الفحم و30% من الميتاكاولين. تم خلط هيدروكسيد الصوديوم بتركيز 14 مولاري مع سيليكات الصوديوم بنسبة 2.5:1 لتحضير المحلول المفعل. تم تسليح مونة الجيوبوليمر باستخدام الياف قشور الرز بنسب (1%, 1.5%, 2%) ,وأيضا تم إضافة مخلفات الورق (عجينة الورق ورماد الورق) بنسب (1%, 2%, 5%) من حجم المواد السمنتية. تم أنضاج عينات مونة الجيوبوليمر بدرجة حرارة 60° سيليزية بالفرن لمدة 24 ساعة. أظهرت النتائج إن اعلى زيادة في مقاومة الانضغاط تم الحصول عليها هي (59% و 55%) للعينات المسلحة بألياف قشور الرز بنسبة 2% والتي انضجت لمدة 7 و28 يوم على التوالي. بشكل عام, إضافة المخلفات الورقية و الياف قشور الرز الى مونة الجيوبوليمر عملت على تحسين مقاومة الانضغاط بشكل ملحوظ.

الكلمات المفتاحية: مونة الجيوبوليمر, رماد الفحم, ميتاكاولين, مقاومة الانضغاط, مخلفات ورقية, الياف قشور الرز.

1. INTRODUCTION

One ton of CO_2 is emitted in one ton of clinker production in cement manufacture, accounting for 7% of worldwide CO_2 emissions. As a result, it is vital to analyze the technological and physical components used in concrete preparation that are eco-friendly and decrease CO_2 emissions **(Bisarya et al., 2015; Muhsin and Fawzi, 2021a; Hardjito and Rangan, 2005)**. The reduction of CO_2 emissions in cement manufacturing and the promotion of recycling industrial waste, which significantly contributes to environmental pollution, can be achieved through the incorporation of sustainable materials in civil engineering projects **(Muhsin and Fawzi, 2021b; Hussain and Aljalawi, 2022)**. In an attempt to produce concrete and mortar, many materials have been substituted for cement, such as Rice Husk, Fly Ash, Silica Fume, Metakaolin, and Ground Granulated Blast Furnace Slag (GGBFS). Additionally, the development of nano-concrete has been explored, involving the integration of carbon nanotubes or self-sensing carbon nanotubes into the concrete mixture to enhance its strength, stiffness, and durability **(Duxson et al., 2007; Popov et al., 2021; Suhendro, 2014; Phan and Nguyen, 2021)**. Davidovits introduced the term "geopolymer" in 1978 in order to designate a class of mineral binders that display chemical characteristics similar to those of zeolites. But unlike Portland cement, geopolymers generate strength by the polycondensing of silica and alumina precursors **(Provis and Van Deventer, 2009)**. Geopolymer concrete has recently emerged as an acceptable substitute for traditional concrete. The primary limitations holding back the widespread acceptance of geopolymer concrete are the lack of established protocols and concern about its long-term durability **(McLellan et al., 2011; Nuruddin et al., 2016; Chouksey et al., 2022; Radina et al., 2023).**

The effect of sodium hydroxide to sodium silicate ratio on the geopolymer concrete has been extensively examined by many researchers. They concluded that the properties of geopolymer concrete appear to deteriorate as the ratio of sodium hydroxide to sodium silicate increases. Increasing the concentration of sodium hydroxide from 8 to 16 M has the potential to enhance the compression and absorption of geopolymer concrete **(Mathew and Issac, 2022; Aliabdo et al., 2016; Kupaei et al., 2014)**. The incorporation of sustainable fibers in both conventional and geopolymer concrete has been found to enhance numerous mechanical characteristics, such as compressive strength, split tensile strength, dry density, and flexural strength. Moreover, it has been discovered that integrating this element decreases water absorption **(Hussain and Fawzi, 2022; Hussein, 2021a)**.

(Hussein, 2021b) examined various ratios of slag to fly ash, namely 25:75%, 35:65%, and 45:55%). The results indicated that the combination of 45% blast furnace slag and 55% fly ash produced the highest strength readings. Furthermore, this investigation showed that the previous mixture ratio might provide a viable solution to the heat-curing requirement for geopolymer materials made from fly ash. The strength and impact resistance of fly ash-GGBS geopolymer concrete both improve as the NaOH solution molarity increases. In addition, reinforcement fibers enhance energy absorption in the first stages of crack development and failure **(Kiran et al., 2015; Amouri and Fawzi, 2022)**. Substituting metakaolin for fly ash in the production of geopolymers has been demonstrated to mitigate drying shrinkage and address structural stability concerns in the final products. The study revealed a linear reduction in apparent porosity and water absorption values with increasing levels of metakaolin substitution, additionally, the effectiveness of the geopolymer declined as more fly ash was replaced by metakaolin, while its strength increased **(Duan, 2016; Gorhan et al., 2016; Yang et al., 2017)**.

(Kareem, 2012) studied the effect of using waste paper fibers on the fresh and some hardened properties of concrete. The results showed that adding waste paper increases the compressive strength and the flexural strength of concrete by (34.21%) and (42.4%) respectively, at 90 days of curing when (1%) of waste paper was added by volume of concrete. **(Zaki et al., 2019)** examined the effect of adding waste paper in (5%, 10%, 15% and 20%) by volume of cement, on some mechanical properties of cement mortar like (compressive, direct tensile, and flexural) strength and thermal conductivity. The results showed that the mechanical properties (compression, direct tensile and flexural strength) were reduced by increasing the content of waste paper by (74%, 50%, and 86%) respectively, with (20%) addition of waste paper in comparison to the reference mix. Also adding waste paper led to a noticeable decrease in thermal conductivity, especially with increasing addition percentages. **(Qasim and Aljalawi, 2023)** conducted a study about the effect of incorporating 1% of rice husk fibers on the slump, dry density, and compressive strength of reactive powder concrete (RPC). In contrast to the control mixture, the compressive strength of RPC exhibited a 7.4% increase, the dry density experienced a decrease of 0.69%, and the workability witnessed a decrease of 5.62%.

The main objective is to lower the environmental impact of cement production by lowering carbon dioxide emissions while developing high-quality, reasonably priced concrete. Therefore, this research investigated the compressive strength of fly ash-metakaolin geopolymer mortar to advance our comprehension of the characteristics and effectiveness of structural geopolymer mortar. In addition to highlighting the possibility of using rice husk fibers, a sustainable material, and waste paper ash as a filler, there are few published works on this subject.

2. EXPERIMENTAL WORK

2.1 Materials

2.1.1 Fly ash

The classification of fly ash as class F is determined based on the criteria specified in **ASTM C618, 2019**, as presented in **Tables 1** and **2.** The used fly ash exhibits pozzolanic properties.

Table 1. Chemical Requirements of Fly Ash Comparing with ASTM C618-19

Oxide	Contents, %	(ASTM C618-19) Requirement
Fe_2O_3	5.31	Total Sum. > 50%
Al_2O_3	18	
SiO_2	64.73	
SO_3	0.25	Maximum 5%
MgO	0.87	--
CaO	2.3	Maximum 18%
K_2O	3.52	--
Na_2O	2.22	--
L.O.I	2.8	Maximum 6%

Table 2. Physical Properties of Fly Ash

Physical property	Results	(ASTM C618, 2019) Requirement
Specific gravity	2.21	--
Physical form	Powder	--
Surface area m^2/kg	780	--
Color	Grey	--
Amount retained when wet sieved on 45 μm (No. 325)%	20	Max 34%

2.1.2 Metakaolin

N-metakaolin is classified as a natural pozzolana that satisfies the specifications outlined in **(ASTM C618, 2019)**, as indicated in **Tables 3** and **4**.

2.1.3 Sodium Hydroxide (NaOH)

The process involves melting caustic soda flakes with purified water to generate sodium hydroxide with a purity level of 99%. The ratio between water and soda flakes can determine various molar concentrations. The **(ASTM E291, 2009)** standard provides further detail about the NaOH solution. In this investigation, 14 M NaOH was used.

Table 3. Chemical Requirements of Metakaolin Comparing with **(ASTM C618, 2019)**

Oxide	Contents, %	(ASTM C618, 2019) Requirement
Fe_2O_3	1.93	Total sum.> 70%
Al_2O_3	40.45	
SiO_2	51.4	
SO_3	0.32	Maximum 4%
MgO	0.47	--
CaO	2.3	Maximum 18%
K_2O	0.272	--
Na_2O	1.018	--
L.O.I	1.84	Maximum 10%

Table 4. Physical Properties of Metakaolin

Physical property	Results	(ASTM C618, 2019) Requirement
Specific gravity	2.54	--
Physical form	powder	--
Surface area m^2/kg	1750	--
Color	Off white	--
Amount retained when wet sieved on 45 µm (No. 325)%	24	Max 34%

2.1.4 Sodium Silicate (Na_2SiO_3)

The sodium silicate employed was produced in the UAE, and its concentration was determined using the Na_2O to SiO_2 and H_2O ratio.

2.1.5 Water

The water used for mixing is complied with **(IQS No.1703, 2018)**.

2.1.6 Fine Aggregate

Natural sand was the fine aggregate used in this experiment. It was categorized as Zone Two and satisfied the Iraqi standard's chemical and physical requirements **(IQS No.45, 2019)**. This compliance is evidenced by the data presented in **Tables 5** and **6**. The sand used is saturated surface dry.

2.1.7 Wastepaper

The microstructure of the paper determined the characteristics of the wastepaper. The primary material used in this research was a wastepaper (printing paper) obtained from offices, schools, and universities.

Table 5. Sieve Analysis of the Fine Aggregates

Sieve No.	Accumulative passing %	(IQS No.45, 2019) Zone Two
10	100	100
4.75	96	90-100
3	84	75-100
1.18	69	55-90
0.6	50	35-59
0.3	15	8-30
0.15	2	0-10

Table 6. Physical and Chemical Properties of the Fine Aggregates

Physical properties	Test results	(IQS No.45, 2019)
Specific gravity	2.67	--
Fineness modulus	2.84	--
Density (kg/m^3)	1590	--
Absorption %	1.8	--
Sulfate content	0.14	0.5% max.

2.1.7.1 Wastepaper Ash

The papers were cut into small pieces with 8*2*0.1 mm dimensions. Then, it was burned in a furnace at 525°C. After burning, the ash was sieved at 600 μm. Ash wastepaper was used as a (1%, 2%, and 5% by volume) addition to the cementitious materials. The oxidation content of wastepaper ash is (20 SiO_2; 4.2 Al_2O_3; 0.76 Fe_2O_3; 78.74 CaO; 0.88 MgO %). The specific gravity of paper ash is 2.4, and L.O.I. is 5.2%.

2.1.7.2 Paper Pulp

A paper shredder cuts waste papers into little shreds with 8 * 2 * 0.1 mm dimensions. These small dimensions prevent clumping when the wastepaper is mixed with water, resulting in equally dispersed papers in paper pulp mixtures. The papers were then soaked for three consecutive days in room-temperature water to stop them from absorbing the water used for mixing. The paper sludge was then placed on a nonabsorbent surface to remove excess water before being pulverized with a kitchen mixer to the required size. The oxide content of paper pulp is (10 SiO_2; 3.2 Al_2O_3; 0.2 Fe_2O_3; 80.74 CaO %).

2.1.8 Rice Husk Fibers

The diameter of the rice husk fibers is 0.5 mm, the length is 5 mm, the density is 700 kg/m3, and the aspect ratio is 10.

2.1.9 Super Plasticizer

BETONAC-1055, a locally sourced product, was used in this research. It complied with the (**ASTM C494/494M, 2005)** type F standard and was utilized to improve the workability of the mix.

2.2 Mixing

It is necessary to prepare the NaOH solution one day in advance of the intended mixing. The sodium hydroxide (NaOH) concentration used in the geopolymer mortar was established at a molarity of 14 M. The ratio of Na_2SiO_3 (161 kg/m^3) to NaOH (41 kg/m^3) was set as 2.5:1, the water added was 10% of cementitious material, and the S.P. was 1.5%. The ratio of solution to cementitious material was determined to be 0.55. The initial phase in the mixing protocol was combining NaOH solution with Na_2SiO_3 nanoparticle, 1.5% solid particles, and 10% water for 2–5 minutes. Subsequently, the dry constituents, namely 70% fly ash (287 kg/m^3), 30% metakaolin (123 kg/m^3), and sand (1476 kg/m^3) in a saturated surface dry (SSD) state, were meticulously blended manually for around 5 minutes. Subsequently, a proportion of 75% of the liquid materials was added to the dry materials, followed by manual mixing for an additional duration of 10-15 minutes. The mixture was then allowed to rest for 15 seconds before adding 25% of the liquid, which was subsequently combined for 10 minutes. Homogeneity was achieved after an approximate duration of 30 minutes of mixing. **Table 7** illustrates the mixed proportions of geopolymer mortar.

Table 7. Mix Design of Geopolymer Mortar for one m^3, Weight in kg/m^3

Mix	RH Fibers% by vol	Paper Ash %by vol	Paper Pulp % by vol
GPr	-	-	-
GP1	1	-	-
GP2	1.5	-	-
GP3	2	-	-
GP4	-	1	-
GP5	-	2	-
GP6	-	5	-
GP7	-	-	1
GP8	-	-	2
GP9	-	-	5

The compressive strength test was conducted at two different curing ages, 7 and 28 days. The samples used in the experiments included one without any fibers or waste paper, three samples with varying percentages (1%, 1.5%, and 2%) of rice husk fibers, three samples with varying rates (1%, 2%, and 5%) of waste paper ash, and three samples with varying percentages (1%, 2%, and 5%) of paper pulp.

2.3 Curing

As indicated in **Fig. 1**, the specimens were subjected to heat to 60°C for 24 hours before being moved to a different oven for cooling and temperature maintenance until the test date.

Figure 1. Compressive strength samples while curing

2.4 Testing

Hand mixing was used to produce the geopolymer and reference specimens. The specimens' compressive strength was measured using a universal testing machine applying the ASTM C109-20 testing technique. The specimens were cast using cubical molds that had dimensions of 50 mm x 50 mm x 50 mm. The samples were tested at 7 and 28 days of curing. The completion of the test occurred with the failure of the concrete specimen, as depicted in **Fig. 2**. Eq. (1) is used to calculate the compressive strength as:

$$fm = \frac{P}{A} \quad (1)$$

where: *fm* is the compressive strength in psi or (MPa), *P* is the total maximum load in lbf or (N), and*A* is the area of loaded surface in^2 or (mm^2).

Figure 2. Compressive strength test

3. RESULTS AND DISCUSSION

Compressive strength is well recognized as a fundamental characteristic of concrete mixes and mortars. Hence, the consistent patterns found in conventional specifications heavily depend on this factor. The test results are presented in **Table 8** and **Figs. 3** to **5**.

Table 8. Compressive Strength Test Results

Mix	Compressive strength (MPa)	
	Age of curing (days)	
	Seven	Twenty-eight
GPr	25.32	27.60
GP1	31.65	33.82
GP2	37.48	39.26
GP3	40.19	42.72
GP4	27.33	30.57
GP5	29.53	32.63
GP6	31.29	34.54
GP7	26.41	29.38
GP8	28.49	31.48
GP9	30.76	33.81

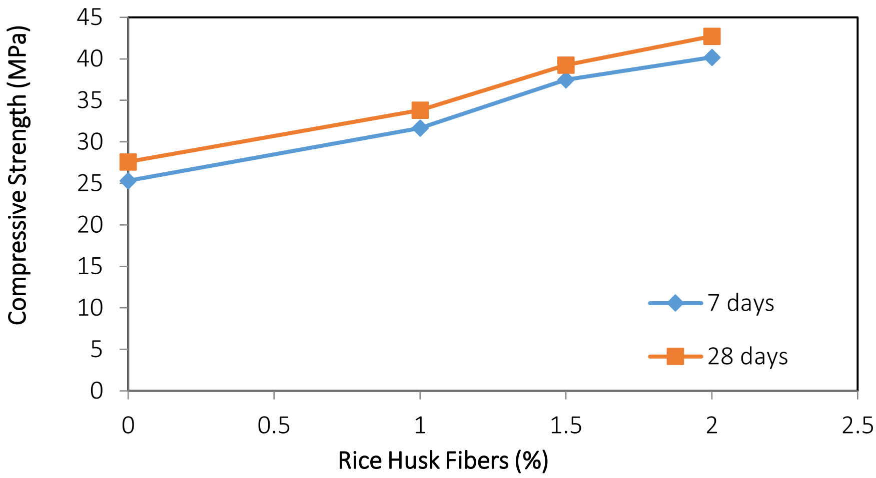

Figure 3. Compressive strength of geopolymer mortar with rice husk fibers at age 7 and 28 of curing

Due to reinforcing the geopolymer mortar with rice husk fibers in (1%, 1.5%, 2%) by volume, the compressive strength increased by (25%, 48%, 59%), respectively, at 7days of curing, while at 28 days of curing, the compressive strength improved by (23%, 42%, 55%). Adding the paper ash in percentages of (1%, 2%, 5%), increased the compressive strength by (8%, 17%, 24%), respectively, at seven days of curing and 28 days, by (11%, 18%, 25%). Moreover, adding paper pulp in (1%, 2%, 5%) by volume of mortar increased the compressive strength by (4%, 13%, 22%), respectively, at age seven days, whereas at 28 days of curing, it increased by (6%, 14%, 23%). The results above show that paper ash results better than paper pulp when used as filler.

The presence of fibers alters the post-crack behavior of concrete. Instead of a sudden failure, fiber-reinforced concrete tends to exhibit a more gradual failure mode. This behavior allows the structure to provide more warning before complete failure, providing an opportunity for repair or maintenance. Also, fibers can enhance the bonding between the mortar particles and other components of concrete. The improvement bonding leads to a more homogenous and stable matrix, resulting in increased compressive strength **(Anggraeni et al., 2022; Qasim and Aljalawi, 2023)**.

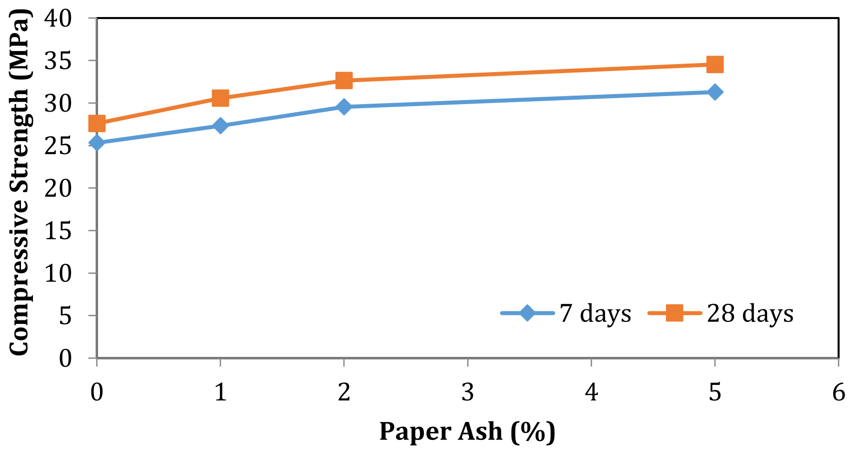

Figure 4. Compressive strength of geopolymer mortar with paper ash at age 7 and 28 days of curing

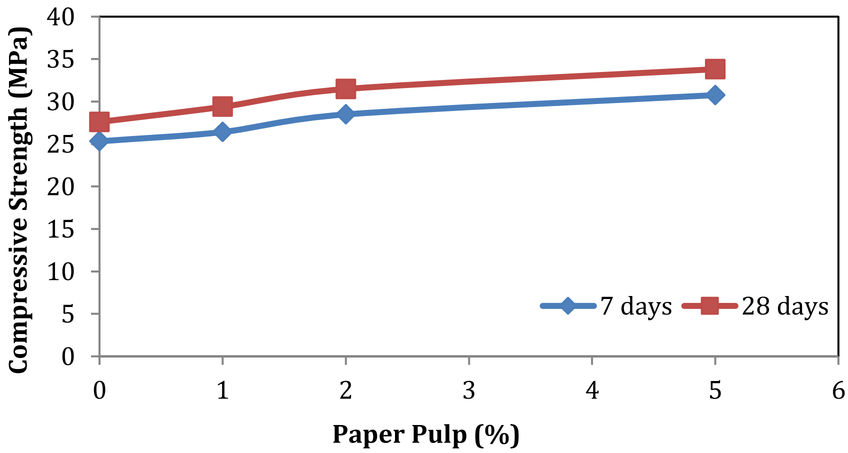

Figure 5. Compressive strength of geopolymer mortar with paper pulp at age 7 and 28 days of curing

The increase in compressive strength on adding paper ash and paper pulp can be explained by the act of the paper ash and paper pulp in filling the pores of the geopolymer mortar, also the products produced during the geo-polymerization process with age lead to preventing the formation of micro-cracks which increases the compressive strength. These results agree with the by **(Al Zubaidi et al., 2019)**.

4. CONCLUSIONS

Based on the compressive strength test results of geopolymer mortar, the following points were concluded:

a. Adding paper pulp in (1%, 2%, and 5%) by volume increased the compressive strength. After 7 days of curing by (4%, 13%, and 22%), and after 28 days of curing by (6%, 14%, and 23%).

b. Adding paper ash in (1%, 2%, and 5%) by volume increased the compressive strength by (8%, 17%, and 24%) at 7 days and (11%, 18%, and 25%) at 28 days.

c. Reinforcing geopolymer with (1%, 1.5%, and 2%) rice husk fibers increased the compressive strength of geopolymer mortar by (25%, 48%, and 59%) after seven days of curing, and (23%, 42%, and 55%) after 28 days of curing.

The results showed that utilizing different percentages of waste paper and rice husk fibers improved the compressive strength of geopolymer mortar.

NOMENCLATURE

Symbole	Description
fm	the compressive strength, MPa
P	the total maximum load, N
A	the area of loaded surface, mm^2

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Credit Authorship Contribution Statement

Sally Hashim: writing original draft, validation, methodology.
Nada Mahdi Fawzi: review and editing, validation, proofreading.

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.

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