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EFFECT OF ALKALI-ACTIVATION ON SETTING CHARACTERISTICS OF BINARY AND TERNARY BLENDED GEOPOLYMER MORTAR

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Abstract

To achieve sustainability, efforts have beared to eliminate the problem connected with cement usage and production by completely replacing cement with an environmentally benign binder. This study investigates the impact of alkali activation on the setting properties of three precursors (Metakaolin, Cassava Peel Ash, and Rice Husk Ash) combined for binary and ternary geopolymer mortar. The fresh properties of geopolymer mortars, including flowability, setting time, and strength, were studied. For each test, a binary mortar mix ratio of CPA/MK, MK/RHA, and CPA/RHA, such as 100/0, 0/100, 75/25, 50/50, and 25/75, was assessed, as well as a ternary mortar mix ratio of MK/CPA/RHA, such as 100/100/100, 50/25/25, 25/50/25, and 25/25/50, and 100%PC (Control). As activators for geopolymertar mixture, 6M, 9M, and 12M concentrations of Na₂SiO₃/NaOH solutions were utilized. The results demonstrated that the control mortar containing 100%PC flowed less than the binary and ternary mortars. Mixtures with a greater proportion of CPA cure faster than those containing a greater proportion of MK or RHA. In addition, the water-to-binder absorption rate of RHA is greater than that of MK and CPA. On the basis of its soundness features, the 100%PC (control) exhibits less expansion than the binary and ternary blended geopolymer mixtures. As an environmentally friendly building material, geopolymer mortar has shown great feasibility and application potential, according to the current study.

Keywords: Alkaline Activation; Cassava peel ash; Geopolymer; Metakaolin; Rice husk ash

1.0 Introduction

Construction companies are under intense pressure to use a green strategy while building infrastructure as we go into the twenty-first century (Obonyo et al., 2011). Concerns are raised about the introduction of hazardous pollutants into the atmosphere, which have the potential to produce greenhouse effects and according to Yoro et al. (2020), major sections generating these toxic gases (such as CO₂, CH₄, N₂O, and so on) include refineries, steel industry, transportation, power generation, and construction industries to mention a few. As a result, steps are being taken to address the growing worry over global warming, which is being produced by numerous industrial sectors. The usage of unconventional materials in place of Portland cement has arisen from the search for more durable and environmentally friendly blended materials that are less prone to cracking. The most widely utilized binding material is still cement, which was one of the first materials employed in cementitious construction materials (Sumesh et al., 2017). According to studies, one ton of Portland cement emits about one ton of CO2 as greenhouse gases. This suggests that a large number of gaseous chemicals are released during the production of cement. (Mohamad et al., 2021). Owing to this fact, several efforts have been made by researchers to reduce the challenge faced when using cement in construction, in which case its quantity can be substituted with nonconventional naterials such as Rice Husk Ash, Flag Ash, Granulated Blast Furnace Slag, Cassava Peel Ash and Metakaolin (Liew et al., 2017). In light of this, it is necessary to create a novel composite material that may be used in place of traditional cement in the construction sector. According to Sambucci et al. (2021), geopolymer binders obtained from pozzolans have attracted the interest of many researchers in the past decades, although the application of such binders for construction purposes is not a new concept. However, attention has been called to a new class of binders for construction applications in which a binder with boosting qualities is greatly desired. This is because of the influence of greenhouse gas emissions linked with the production of cement. (Kumar& Mittal, 2019). For the creation of sustainable target-oriented geopolymer materials, several agricultural waste products have been used, including wood fibers, recycled newspaper, recycled brown papers, potato peels, rice husk ash, and cassava peels, among others. However, cassava peel ash proves to be one of the most interesting renewable agricultural waste products due to its availability and ease of production because the ash from cassava peels contains silica and alumina, it also offers sustainability in terms of effectiveness, durability, and environmental friendliness. (Ofuyatan et al., 2018) However, rice husk (RH), which makes up about 20% of rice, is a readily available agricultural byproduct in underdeveloped nations, leading to disposal and pollution issues. RH is burned to produce RHA, which has a silica content of roughly 90% (Sekou et al., 2017). RHA is made of highly reactive Pozzolan and non-crystallin silicon oxide (SiO2) with a high specific surface area. To improve the strength and durability of mortar, additional cementitious ingredients like Rice Husk Ash are included. The fortifying agent sodium silicate, which is an oxide of sodium and silica, is typically used with sodium hydroxideor potassium hydroxide to increase alkalinity and, in particular, material strength (Giacobello et al., 2022). Therefore, it is important to mention that incorporation of sodium silicate and Sodium Hydroxide would stand a better chance as an activator in geopolymer mortas. On this note, an alternative binder with full replacement of cement with Metakaolin and agricultural waste geopolymer (particularly Cassava Peel Ash and Rice Husk Ash), would restrain the level of mining and offer environmental benefit in terms of waste recycling. Therefore, this study is focused on the development and application of geopolymer mortar consisting of a ternary mixture of Meta-kaolin, Cassava Peel Ash, and

Rice Husk Ash. Further to ascertain the activation potential of the effect of Na₂SiO₃/NaOH on the prepared binary and ternary blended geopolymer mortars and to investigate the setting properties of the produced mortar. The use of such wastes as sustainable construction materials takes care of the issues of environmental pollution (Raheem & Ikotun, 2020).

2.0 Materials and Methods

Metakaolin, Cassava peel ash and Rice husk ash are the three solids (precursors) useith this study (Figure 1), fine aggregate, the alkaline liquid used are NaOH in 6, 9 and 12 Molar concentration, Na₂SiO₃, water, and superplasticizer the materials were obtained from nearby local source. The chemical composition of all ashes was presented in Table 1 while the detailed mixture design and mixture proportions are given in Table 2, 3 and 4. The setting time and the flowability test for the Binary (MK/CPA, MK/RHA, CPA/RHA) and Ternary (MK/CPA/RHA) studies of geopolymer mixtures in a combination proportion of 0%, 25%, 50%, 75%, and 100% to replacement of cement. A Binder of 140g was used in preparing the flowability and 300g of binder was used in carrying out the setting time. The Alkaline to binder ratio used is ranged from 0.5 to 0.8 and the ratio of NaOH to Na₂SiO₃ was 1:2.5 in preparing the mix.



Figure 1: Powered sample of (a) RHA (b) CPA and(c) MK

Table 1: Chemical compositions of CPA MK and RHA (mass%)

Materials	SiO ₂	Al_2O_3	CaO	SO3	F	e_2O_3	Na ₂ O	K ₂ O	TiO ₂	MnO
MgO LOI										
MK 2.35	72.39	20.35	0.01	-	1.12	0.34	3.12	0.90	0.02	0.12
CPA 2.10	80.83	0.77	4.24	0.83	1.55	0.06	5.50	-	0.05	-
RHA 0.74	48.62	5.20	3.20	8.75	3.25	0.01	10.2	0.02	0.66	0.45

Source: Experimental work (2021)

Figure 2 illustrate the X-Ray Diffraction (XRD) patterns of CPA, MK and RHA. The XRD pattern of CPA revealed a pronounced broad hump with diffraction peaks at 2α values in the range of $27\text{-}56^\circ$, few sharp crystalline diffraction peaks indicate its dominant amorphous phase and crystalline phases of 111,220,330. The XRD pattern of MK demonstrates an outstanding crystalline phase material with obvious detectable quantities of kaolinites and silica while XRD pattern of RHA showed an amorphous phase with a diffraction peaks at 2α values in the range of

20-25° with a few crystalline diffraction peak. Figure 3 displays the SEM images of CPA MK and RHA used in this study. The surfacephology of CPA clearly revealed gelatinous appearance with irregular globular shaped particles as shown in Figure 3(a), whereas MK manifested irregular pellet-like and angular particles arranged disorderly Figure 3(b). RHA appeared clumsy with irregular circular shape with few stretchy threads like particles as shown in Figure 3(c)

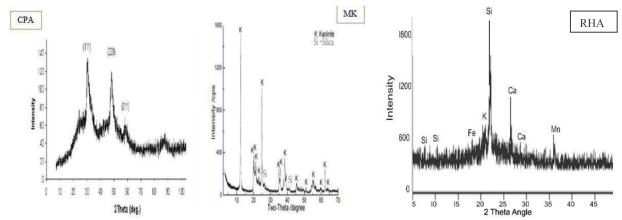


Figure 2: XRD of (a) CPA, (b) MK and (c) RHA

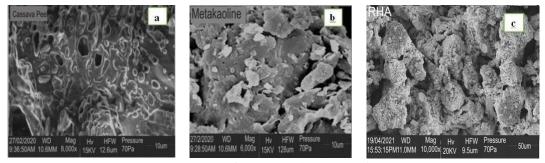


Figure 3: SEM of (a) CPA (b) MK and (c) RHA

2.1 Alkali solution

A mixture of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH, purity 98%) alkaline solution used in the present study (Figure 4). These were used to activate the alumina and silica in MK and CPA. The Na_2SiO_3 solution was composed of SiO_2 (27.0 mass %), Na_2O (14.0 mass %) and H_2O (51.0 mass %). These chemicals were purchased from Niger State. A different amount of pellet was dissolved in water to prepare NaOH solution of various molar concentrations (6, 9 and 12M). The solution was left for 24h to be cool because when it was

mixed, it started to react thereby emitting a large amount of heat and for this reason; the heated solution was allowed to cool at room temperature before use then it was added to a Na₂SiO₃ solution to prepare the final alkaline solution. The ratio of sodium silicate to sodium hydroxide (NS: NH) was 1:2.5 fixed for all mixtures of alkaline solution. Table 2 illustrate the details of the alkaline solution composition



Figure 4: Images of NaOH and Na₂SIO₃

Table 2: Composition of Alkaline Solution

Alkaline S mass%	Solution	NaOH solution (N	NH)	Na2	NS:NH		
	Molarity l	M Na20 mass%	H2O mass%	SiO2 mass%	Na20 mass%	H20 mass%	
S1	6	18.6	81.4	27.0	14.0	59.0	2.5
S2	9	27.9	72.1	27.0	14.0	59.0	2.5
S3	12	37.2	62.8	27.0	14.0	59.0	2.5

2.2 Method of Mix

Geopolymer mortar is a mixture of any supplementary materials as binder, sand and fluid (sodium hydroxide, sodium silicate, superplastisiær and water). Metakaolin, Rice Husk ash and Cassava Peel ash in its original form cannot function as binder rather it can be used just as filler material in cement mortar as a replacement of cement. However, the above binders can be activated by incorporating a strong alkali solution of sodium hydroxide and sodium silicate. The activated above said binders which is rich in silica and aluminium can function as a binder like Portland cement. The geopolymer mortar was prepared using water to solid ratio (w/s) 0.27, 0.26 and 0.25. The water content is the total water in activator and additional water whilst the solid is the CPA MK and RHA and solid part of the activator. A Binder of 140g was used in preparing the flowability and 300g of binder was used in carrying out the setting time. The Alkaline to binder ratio used is ranged from 0.5 to 0.8 and the ratio of NaOH to Na₂SiO₃ was 1:2.5 in preparing the mix. Thus, investigation of geopolymer mortar excluding RHA alkaline liquid to binder ratio of 0.5 and geopolymer mortar including RHA alkaline liquid to binder ratio of 0.8 was used and the mix proportions and details of mix are given in Table 3,4 and 5 respectively.

Table 3: Consistency and Setting Time Mix Design for Binary and Ternary Blended Mortar

Mix	Тур	es of B	inder	Alkalir	ne Solution	Consistency/ B	
	MK	СР	RHA	NS:N	Molarity	33% - 45%	
		A		Н	1110101110		
	0	100	_	2.5:1	6, 9, & 12	40%	
	50	50	-	2.5:1	6, 9, & 12	40%	
M1	75	25	-	2.5:1	6, 9, & 12	33%	
	100	0	-	2.5:1	6, 9, & 12	33%	
	0	-	100	2.5:1	6, 9, & 12	45%	
	50	-	50	2.5:1	6, 9, & 12	43%	
	75	-	25	2.5:1	6, 9, & 12	35%	
M2	100	-	0	2.5:1	6, 9, & 12	33%	
	-	0	100	2.5:1	6, 9, & 12	45%	
	-	50	50	2.5:1	6, 9, & 12	45%	
	-	75	25	2.5:1	6, 9, & 12	45%	
M3	-	100	0	2.5:1	6, 9, & 12	40%	
	50	25	25	2.5:1	6, 9, & 12	34%	
	25	50	25	2.5:1	6, 9, & 12	38%	
	25	25	50	2.5:1	6, 9, & 12	40%	
M4	0	25	75	2.5:1	6, 9, & 12	45%	

Table 4: Flowability Mix Design for Binary and Ternary Blended Mortar

Mix	Typ	es of B	inder		Alkaline	Solution	•	Super	
ID	MK	CPA	RHA		NS:NH	Molarity	S:B	plasticize	W/B
				Sand				r (%)	
	0	100	-	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.5
	25	75	-	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.5
M1	50	50	-	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.5
	75	25	-	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.5
	100	0	-	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.5
	0	-	100	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	25	-	75	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
M2	50	-	50	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	75	-	25	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	100	-	0	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	-	0	100	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	-	25	75	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
M3	-	50	50	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	-	75	25	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	-	100	0	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	50	25	25	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
M4	25	50	25	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8
	25	25	50	350	2.5:1	6, 9, 12	0.5	2, 5, 10	0.8

Table 5: Flowability of Binary blended and Ternary mix proportion (g/m³)

	Table 5: Flowability of Binary blended and Ternary mix proportion (g/m³)												
Mix	Designation	CEM	CPA	RHA	\mathbf{M}	San	Alkalin	Extra					
ID		(g)	(g)	(g)	K	d	e	Water					
					(g)	(g)		(ml)					
							Solution						
							(ml)						
M1	PC 100% (control)	140	-	-	-	350	70	70					
M2	0%MK 100%CPA	-	140	-	-	350	70	70					
	25%MK 75%CPA	-	105	-	35	350	70	70					
	50%MK 50%CPA	-	70	-	70	350	70	70					
	75%MK 25%CPA	-	35	-	105	350	70	70					
	100%MK 0%CPA	-	-	-	140	350	70	70					
M3	0%MK 100%RHA	-	-	140	-	350	70	112					
	25%MK 75%RHA	-	_	105	35	350	70	112					
	50%MK 50%RHA	_	_	70	70	350	70	112					
	75%MK 25%RHA	_	_	35	105	350	70	112					
	100%MK 0%RHA	_	_	_	140	350	70	112					
M4	0%CPA100%RHA	_	_	140	-	350	70	112					
	25%CPA75%RHA	_	35	105	-	350	70	112					
	50%CPA50%RHA	-	70	70	-	350	70	112					
	75%/CPA/25%RHA	-	105	35	-	350	70	112					
	100%CPA0%RHA	_	140	-	_	350	70	112					
M5	50%MK/25%CPA/25%	_	35	35	70	350	70	112					
	RHA												
M6	25%MK/50%CPA/25%	_	70	35	35	350	70	112					
	RHA												
M7	25%MK/25%CPA/50%	_	70	35	35	350	70	112					
	RHA												

3.0 Results and Discussion

Three molar concentration of NaOH 6, 9 and 12M were designed to study the effect of the activator on binary and ternary blended geopolymer mortar in terms of workability and setting time of mortars. The results were compared for both binary and ternary variation of MK, CPA and RHA at different ratio at a time.

3.1 Workability

The workability of fresh geopolymer mortar mixtures were tested by flow test. The flow of fresh geopolymer mortars was measured in accordance with ASTM C1437 (2007). A flow test was conducted immediately after mixing. Figure 3, 4 and 5 presents the results of the geopolymer mortars flow according to the sodium hydroxide concentration for varying proportion of the constituent materials and several contents of superplasticizer. The results show that flowability of

the mortar tend to increase as the molar concentration of NaOH is increased. Furthermore, geopolymer mortars with superplaticizer showed higher flow compared to mixtures without superplasticizer. It implies that mortars with increase superplasticizer content show an increase flow. This increment could be attributed to the retardation properties of the superplasticizer employed during the mortar mix. Moreover, flowability result of mortar with 100% PC as control is lower in relation to the geopolymer mortar mix. Figure 5 and 6 illustrate the results of the binary blend mix with and without SP and Figure 7 illustrate the result of ternary blend mix with and without SP. Similarly, the flowability the mortar tends to increase as the molar concentration of NaOH is increased. However, the result of the binary blended shows better flowability compared to ternary blended. Therefore, it can be deduced that the increment of sand to ash ratio rarely caused a reasonable change in flowability (Jumrat *et al.*, (2011).

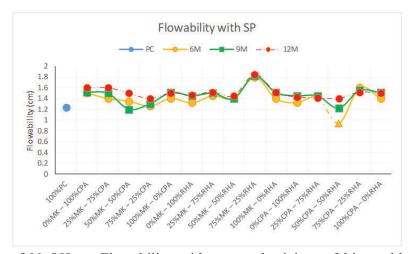


Figure 5:Effect of NaOH on Flowability with superp lasticizer of binary blended mortar containing various percentages of CPA, MK and RHA.

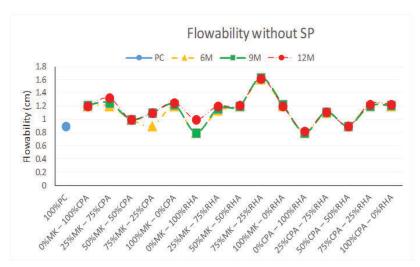
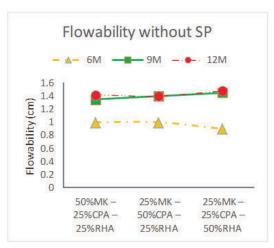


Figure 6: Effect of NaOH on Flowability without superplasticizer of binary blended mortar containing various percentages of CPA, MK and RHA.



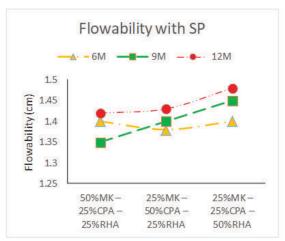


Figure 7: Effect of NaOH on Flowability without superplasticizer and with superplasticizer for ternary blended mortar containing various percentages of CPA, MK and RHA

3.2 Consistency, Initial, Final Setting Time and Soundness.

The standard consistency of the mixtures ranges from 33-35 mm of the plunger penetration. Table 6, 7 and 8 illustrate the effect of different molarity of NaOH on setting characteristics of geopolymer mortar. It can be observed that the effect of NaOH and Na₂SiO₃ prolong the setting time of the binary and ternary binders when compared with control value. However, the results of the various mixtures containing CPA, MK and RHA with 6M, 9M, and 12M of NaOH shows that the higher the molar concentration of NaOH, the longer time it takes for the samples to set. Mortar samples with RHA, MK and RHA/MK showed a longer initial and final setting time while mortar mixtures contain CPA showed speedy initial and final setting time. The present study is in accordance with the report of Saloma et al. (2016). It was stated that an increase in molarity of NaOH can remarkably decrease the itinal setting time as well as the final setting time of geopolymer mortar. Malkawi et al., (2016) concluded that the content of Na is the main factor affecting the setting time because the ratio of Na to Si can be changed by the changing of NaOH molarity. The soundness test results of Portland cement paste "which was used as a control (100/0%)" showed a lesser expansion as against the binary and the ternary paste geopolymer mix. Irrespective of the various proportion of geopolymer mix, mortars with CPA content had higher expansion rate compared to others. The mix ratio of CPA/MK (100/0%, 75/25%, 50/50%), MK/RHA (100/0%, 75/25%, 50/50%), and CPA/RHA (100/0%, 75/25%, 50/50%), as well as, the ternary ration of MK/CPA/RHA (100/100/100%, 50/25/25%, 25/50/25%, 25/25/50%) complete replacement. The expansion of cement was recorded to be 1.00mm which is less than the maximum value of 10mm conforming to BS EN 196-3:2016, and the proportions containing CPA paste was recorded to be 5mm.

Table 6: Consistency, setting time (initial and final), and soundness result for binary blended mortar

mor		Т. Л	1	TA /F	40-4-1-					
Fine	Mix al Sound	Mo	<u>I.</u>	Ma	terials			_		Initial
rilla	Designation (%)		CPA (g)	MK (g)	RHA	Alkaline solution (ml)	Consis tency (mm)	setting time (min)	setting time (min)	ness (mm)
1	50%MK	6M		150	150	129	33	83	15	343
	50%RHA									
	50%MK	9M		150	150	129	33	82	173	3
	50%RHA									
	50%MK	12M		150	150	129	34	90	193	3
	50%RHA									
2	50%MK	6M	150	150		120	34	42	69	3.5
	50%CPA	03.5	4.50	4 = 0		1.00		4.0	0.0	
	50%MK	9M	150	150		120	34	49	82	4
	50%CPA	1014	1.50	1.50		120	2.4	40	0.0	4
	50%MK 50%CPA	12M	150	150		120	34	49	89	4
3	50%CPA 50%CPA	6M	150		150	135	33	52	83	3.2
3	50%CPA 50%RHA	OIVI	130		130	133	33	32	03	3.2
	50%CPA	9M	150		150	135	34	49	85	3.6
	50%RHA	J1V1	150		150	133	54	77	0.5	5.0
	50%CPA	12M	150		150	135	33	57	102	4
	50%RHA									
4	75%MK	6M		225	75	105	34	111	150	2.5
	25%RHA									
	75%MK	9M		225	75	105	33	123	167	3
	25%RHA	103.5		~~~		40.7	2.2	100	100	
	75%MK	12M		225	75	105	33	129	193	3
5	25%RHA 75%MK	6M	75	225		99	34	52	75	3
3	25%CPA	OIVI	13	223))	J T	32	13	3
	75%MK	9M	75	225		99	33	54	89	3
	25%CPA									
	75%MK	12M	75	225		99	33	57	102	4
_	25%CPA	0.5				10.5	2.4	•		
6	75%CPA	6M	225		75	135	34	26	51	4
	25%RHA 75%CPA	9M	225		75	135	34	36	71	4
	75%CPA 25%RHA	71VI	223		13	133	34	30	/ 1	4
	75%CPA	12M	225		75	135	33	49	79	5
	25%RHA				, =					

 $\textbf{Table 7}: \textbf{Consistency, setting time (initial and final), and soundness result for ternary blended GP mortar 100\%$

Mix ID	Molarit y		Materia	als	Alkaline solution	Consis tency	Initial settin	Final Settin	Sound ness
10	y	CP A (g)	MK (g)	RHA (g)	(ml)	(mm)	g time (min)	g time (min)	(mm)
	6M			300	135	34	63	148	2
100%RH	9M			300	135	34	86	162	3
A	12M			300	135	34	76	186	2
	6M		300		99	34	76	129	2
100%MK	9M		300		99	33	74	124	3
	12M		300		99	33	81	156	3
	6M	300			120	34	44	79	3
100%CPA	9M	300			120	35	46	79	4
	12M	300			120	35	47	83	4

Table 8: Consistency, Setting Time (initial and final), and Soundness Result for Ternary

Mix	Designation (%)	Mol	•	Matei	ria			Initial setting time	Final setting time	Soun- dness (mm)
			CPA (g)	MK (g)	RHA (g) (ml)	Alkaline soln.	Consiste- ncy (mm)	(min)	g time	(11111)
1	100%PC						33	47	90	1
2	50%MK 25%CPA 25%RHA	6M	75	150	75	102	34	65	105	3
	50%MK 25%CPA 25%RHA	9M	75	150	75	102	34	74	127	3
	50%MK 25%CPA 25%RHA	12M	75	150	75	102	33	78	183	3
3	25%MK 50%CPA 25%RHA	6M	150	75	75	114	35	34	63	4
	25%MK 50%CPA 25%RHA	9M	150	75	75	114	35	48	68	4
	25%MK 50%CPA 25%RHA	12M	150	75	75	114	34	46	75	4
4	25%MK 25%CPA 50%RHA	6M	75	75	150	120	34	63	90	3
	25%MK 25%CPA 50%RHA	9M	75	75	150	120	33	62	102	4
	25%MK 25%CPA 50%RHA	12M	75	75	150	120	33	68	115	4

5	100%MK 100%CPA 100%RHA	6M	100	100	100	120	49	65	34	3
	100%MK 100%CPA 100%RHA	9M	100	100	100	120	52	78	33	4
	100%MK 100%CPA 100%RHA	12M	100	100	100	120	61	93	33	4

4 Conclusion

The study focuses on the effect of sodium hydroxide/sodium silicate on setting characteristics of geopolymer mortar. The environmental benefit of efficiently using these waste materials is the primary motivation for this study. From the observations made and experimental findings, it was seen that the alternative binders from CPA, RHA (Agro waste) in combination with MK (Geological based solid) possess good binding properties. The chemical analysis reveals that the constituent materials can be classified as Class N Pozzolan with high SiO₂ content. It can also be seen that NaOH/Na₂SiO₃ and NaOH molar concentration for 6M, 9M, and 12M has significant effects on the flowability and setting characteristics of the fresh mortar mix. Finally, the current research results indicate that the geopolymer mortar has exhibited significant feasibility and application prospect to be used as an environmentally friendly building material, which may be an appropriate alternative to conventional cement mortar in future.

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