Evolvement of one-part alkali activated fine soil/limestone powder based geopolymer mortar

Arass Omer Mawlod

University of Raparin, Raniyah, Kurdistan Region, Iraq.

arass.omer@uor.edu.krd

Abstract: This study investigates the feasibility of fine soil/limestone powder as geopolymer binder. The fine soil powder was replaced by limestone powder at (0%-100%). The geopolymer mortar cured at room temperature. Solution to binder ratio was kept constant 0.40. The sand content was 20% of the total weight. Mechanical and absorption properties were evaluated. Moreover, the effect of NaOH concentration on the geopolymer properties was also examined. The results show that increasing the limestone content in the mixes accompany the decrease in compressive strength and increase the absorption properties. At the same time increasing the NaOH concentration leads to improve the mechanical properties and decrease the water absorption and water sorptivity. The alkaline solution of NaOH with concentration M14 shows the optimal mechanical and absorption properties.

Keywords: Sodium hydroxide, fine soil, limestone powder, compressive strength, water absorption water sorptivity.

Introduction:

Binders are the active part of the mortar and concrete; ordinary Portland cement is the most commonly used binder in the construction purposes. The manufacturing process of OPC is responsible for about 7% to 8% of CO2 emission to the atmosphere which consider nowadays as a significant global warming [1]. Recently, industrial by-products materials are used as a binder to produce alkali activated concrete with the aid of alkali activators. Alkali activated materials are developed by mixing sodium silicate and sodium hydroxide with waste materials like fly ash [2–4], granulated slag [5,6], metakaolin [7], Ceramic waste [8] and rice husk [9]. Which contain a huge amount of silica and alumina. This activation can minimize the exploitations of unsustainable materials, energy consumption, pollution and the area used to waste landfills, all of that can mitigate the global warming [10]. Currently, sustainability in construction materials is consider a main requirement which prompted researchers to innovate a sustainable construction material as an alternative to conventional concrete. Portland cement production requires a double amount of energy as compared to activator preparation in alkali activated production [11].

The mixture of the sodium hydroxide or potassium hydroxide with sodium silicate or potassium silicate is the common activator utilized for activating alumina-silicate materials [12]. These alkaline solutions are manufactured products. Sodium silicate is produced at high temperature between 1300-1500℃ by melting sand with sodium carbonate [13,14]. This process requires a huge amount of energy and also emits dust, nitrogen oxide and sulphur oxide to the atmosphere [14]. This encourages the researchers to develop one part of alkali activator.

Inclusion of limestone investigated by Qian and Song[15] who replaced Metakaolin with 10-30% of limestone to develop geopolymer paste. Incorporation of limestone deteriorated 1 day mechanical properties, adversely improved the 7 day mechanical properties.

Cwirzen et al[16] replaced metakaolin with 30,50 and 70% of limestone, NaOH was used as alkali activator with 3M and 5M. the results revealed that compressive strength reduced at 28 day for the 3M of NaOH. When 5M of NaOH was utilized, compressive strength was improved with 50% of limestone powder substitution, however compressive strength deteriorated with the 70% of limestone powder replacement.

In addition, the durability of geopolymer and conventional concrete was investigated by Alzeebaree et al. [17]**,** they investigated that the geopolymer had less permeability and best durability than conventional concrete.

Karozou et al. [18] examined soil as a geopolymer base material. Mechanical and physical properties were investigated, it was concluded that earthen materials are good option to utilize as geopolymer base material.

Jitha et al[19] examined to develop soil based block, it was concluded that soil based block can be achieved with more than 5MPa

Although there are some studies concerning the mechanical and durability of one-part alkali activated binders. There are no or rare of researches dealing with comparative investigation on one-part alkali activated fine soil powder blended with limestone powder. This study aims to obtain an economical and environmentally friendly alkali activated fine soil/limestone powders with particle sizes smaller than 300μm. The results conducted through investigate the compressive strength, water absorption and water sorptivity properties of alkali activated fine stone binder blended with variance ratio of limestone powder. In addition, the influence of alkali concentrations on compressive strength and sorptivity of fine soil-based alkali activated mortar was reported.

Methods and materials:

Fine soil: the clay was collected and brought from Mirabag village west of Raniah city, cleaned, dried and sieved such that all the particles passed through sieve 300μm, the particle size distribution is shown in the figure 1. And the chemical composition test XRF is conducted and the test results are shown in the table 1.

Limestone: the limestones were collected and brought from Darbandikhan, cleaned, dried crushed to a fine powder and sieved such that all the particles passed through sieve 300µm, the particle size distribution is shown in the figure 1. And the chemical composition test results are shown in the table 1

Chemical composition of XRF test result of clay Fine soil and limestone

Oxides	SiO2	Al2O3	CaO	Fe2O3	SO3	Na2O	MgO	K2O	Mn
Fine Soil	13.926	4.49	60.277	2.737	0.187	0.762	5.892	0.564	10.262
limestone	3.92	2.74	51.01	0.36	Nil	Nil	0.28	41.56	0

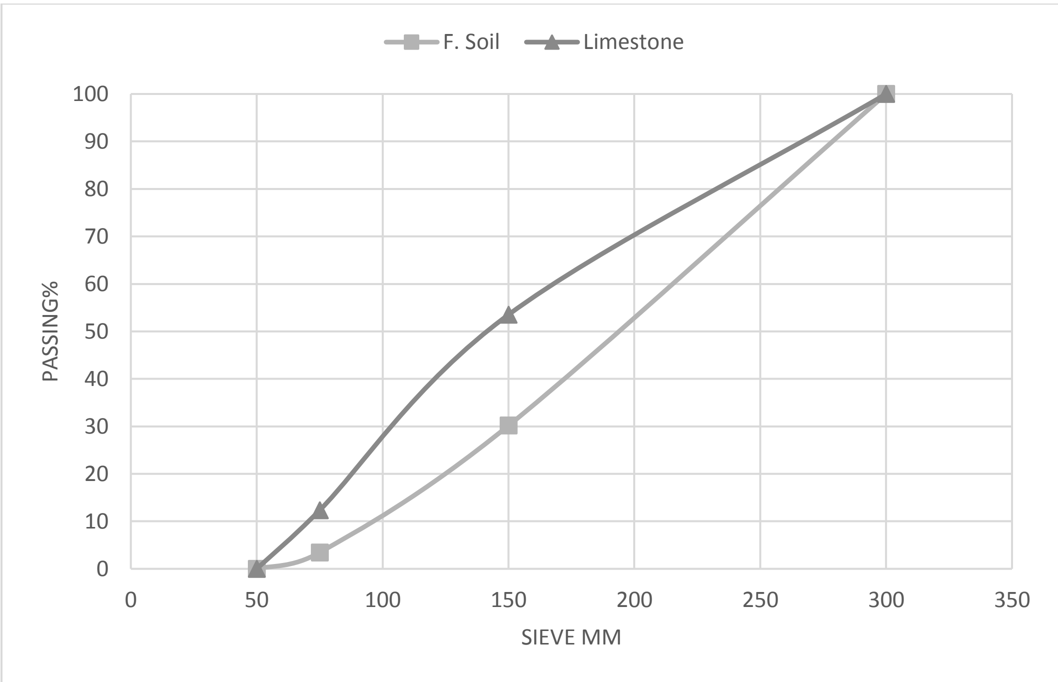


Fig. 1 Particle size distribution of fine soil and limestone powder.

2.1.4 Fine river sand: locally available river sand was used. That is conformed to ASTM C33. Specific gravity is 2.64.

2.1.5 Sodium Hydroxide: Sodium hydroxide in flakes with purity (99%) was used for all of the experimental mixes of the research.

2.1.6 Water: distilled water was used for preparing the NaOH solution for investigation.

Fig. 2. Materials used in the experimentation.

Mix design:

This study contains three series of mixes, first one was five mixtures that was designated of fine soil blended with limestone powder in different ration (0%, 25%, 50%, 75% and 100%). with three different concentration of NaOH solution M10, M12 and M14. to examine the effect of NaOH concentration on compressive strength and absorption properties of the mortar. Fine soil/limestone powder were used as base material. The binder to solution ratio was kept constant 0.40. 20% of the total weight for fine sand was used. The mix proportions are presented in the table 2.

Table 2 Mix Proportion of the geopolymer mortar (Kg/m3)

Mixes	Proportion of binders	Fine Soil	Limestone	Fine Sand	NaOH Molarity	NaOH Solution	Binder/ Solution
M1	S100-L0	1215	0	467	10	486	0.40
M2	S75-L25	911.25	303.75	467	10	486	0.40
M3	S50-L50	607.5	607.5	467	10	486	0.40
M4	S25-L75	303.75	911.25	467	10	486	0.40
M5	S0-L100	0	1215	467	10	486	0.40
M6	S100-L0	1215	0	467	12	486	0.40
M7	S75-L25	911.25	303.75	467	12	486	0.40
M8	S50-L50	607.5	607.5	467	12	486	0.40
M9	S25-L75	303.75	911.25	467	12	486	0.40

M10	S0-L100	0	1215	467	12	486	0.40
M11	S100-L0	1215	0	467	14	486	0.40
M12	S75-L25	911.25	303.75	467	14	486	0.40
M13	S50-L50	607.5	607.5	467	14	486	0.40
M14	S25-L75	303.75	911.25	467	14	486	0.40
M15	S0-L100	0	1215	467	14	486	0.40

Mixing, Casting and Curing:

Sodium hydroxide flakes were dissolved in distilled water to get the require concentration of solution, the binder materials (fine soil partially replaced by limestone powder by (0, 25, 50, 75 and 100)% were blended, with three different concentration of NaOH solution M10, M12 and M14. then mixed with fine sand, the solution added to the dry materials, the mixture poured with two layers into the moulds with size (25*25*25) mm, then each layer was manually compacted to remove the entrapped air. A total of 225 samples were prepared. The samples were covered by a plastic bag to avoid losing of moisture, after 24hr the samples were demoulded and kept at room temperature. The samples were tested for compressive strength at 14, 21 and 28, day, for water absorption and water sorptivity at 28 day of age.

Testing:

1 **Compressive Strength:** is the ability of material to resist failure under the action of compression load. Compressive strength test is conducted according to ASTM C109 for cement mortar. For each mix three samples were tested by a digital compression machine with the capacity of 2000KN. the average of the three results of three samples were calculated and reported. The results of the compression strength of 14, 21 and 28 day of age were presented in the relevant tables and graphs.

Fig. 3 Compression machine test

2 Water absorption: water absorption is the ability of material to absorb water and retain under specific condition. Durability of materials can be evaluated by conducting water absorption test, in this research water absorption test is taken at 28 day of age, for each of the mixes three samples were dried to a constant mass in oven at 105 °C for 24 hr. then the samples kept to cool to room temperature after that the samples were immersed in water for 24 hr to get the saturated mass of the samples, The increase in mass to the dry mass by percentage is the water absorption.

$$\text{WA\%} = \frac{M2 - M1}{M1} * 100$$

M1=dry mass and M2 =saturated mass of the sample

3 Water sorptivity: is the ability of material to absorb water by suction. It is one of the tests related to the durability of the material to evaluate the ingress of water through the material. Water sorptivity of geopolymer mortar was carried out according to ASTM C1585 standard[20]. In this test three samples of (25*25*25) mm were used. Water sorptivity measures the amount of water absorbed by the mortar by suction. In this study for each mix three samples were dried to a constant mass at 105 °C in oven at 28 day, then the samples were taken out, and coated with silicone sealing to avoid entering water from the sides, then the samples kept in water with depth not more than 4mm above the bottom of the samples as shown in the figure 4. Wetted height of the sample can be evaluated by dividing the increase of the mass of the sample weighed at different time intervals, by the bottom surface area of the sample and density of water. Then, the square root of time versus these values was plotted and the sorptivity index of mortar was calculated by the slope of the line of the best fit.

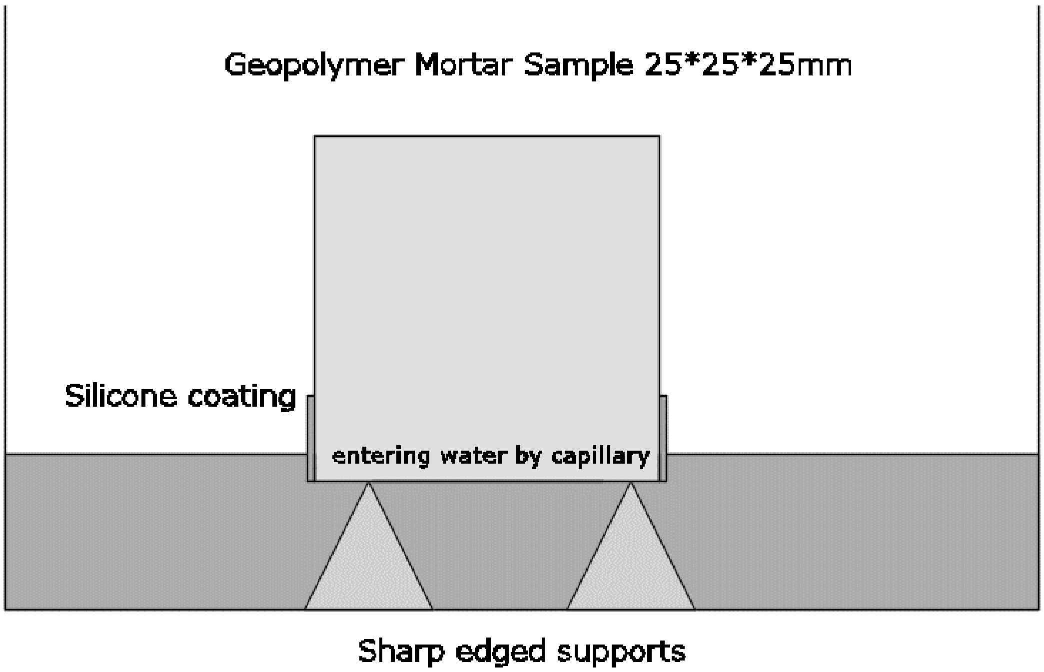


Figure 4 water sorptivity test for geopolymer mortar

3 Discussion:

3.1 Compressive strength:

Table 3and figure 5 show the results of the experimentation conducted to evaluate the effect of limestone powder replacement and changing the NaOH concentration on the compressive strength. It is obvious that with increasing the limestone content from 0% to 100% the compressive strength increases.

At the same time increasing the concentration of NaOH up to 14M compressive strength also increases. This is due to high degree of geopolymerization as a result of increased leaching of alumina and silica. Sakonwan et al. 2014

In the presence of high calcium content, higher molar concentration of NaOH produces higher strength [21]

Alkali solution provides a suitable condition for formation of certain amount of binding gel (C-A-S-H)[22] The unreacted limestone powder particles could also act as filler improving the packing density and creating nucleation sites leading to higher compressive strength[23].

Table 3 Effect of limestone replacement and NaOH concentration variation on compressive strength of fine soil based mortar

Mixes	Compressive Strength (MPa)								
	14 day			21 day			28 day		
	M10	M12	M14	M10	M12	M14	M10	M12	M14
S100L0	3.04	3.46	4.40	3.50	3.76	4.45	4.70	5.75	7.35
S75L25	3.29	4.02	5.29	3.87	4.23	5.50	4.98	6.32	7.43
S50L50	3.98	4.17	6.32	4.89	5.10	6.55	6.33	6.78	7.87
S25L75	4.56	5.10	6.57	5.63	5.90	6.76	6.73	7.03	7.95
S0L100	5.31	5.44	7.07	5.88	6.54	7.14	6.92	7.37	8.33

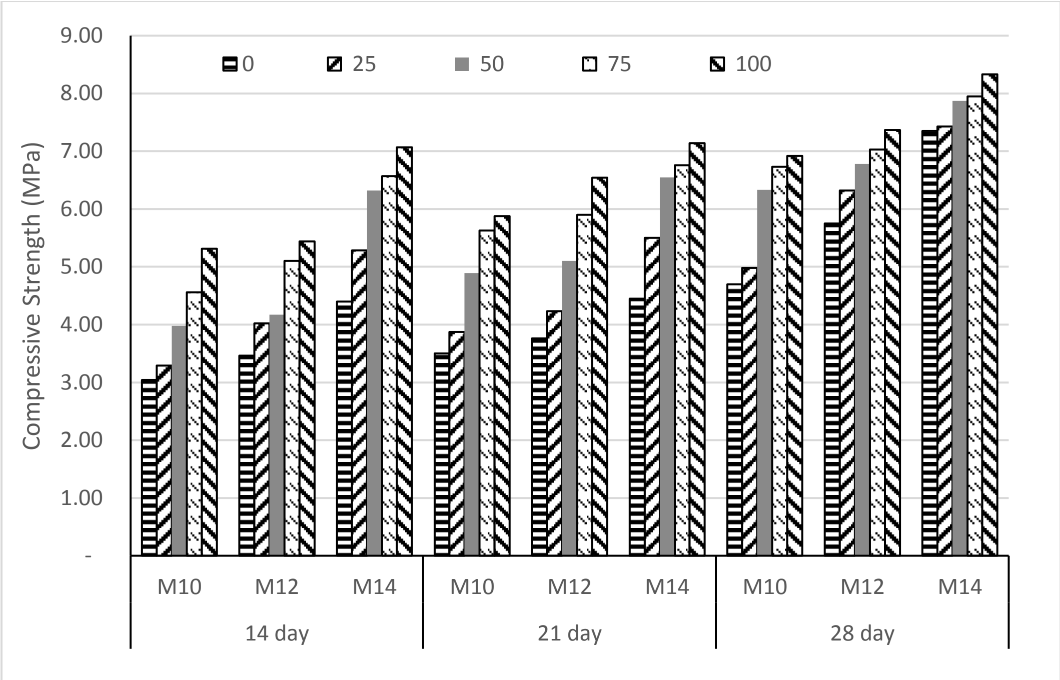


Fig. 5. Effect of limestone replacement and NaOH concentration variation on compressive strength of fine soil based mortar

3.2 Water absorption:

The results of the water absorption are shown in the table 4 and figure 6. Increasing limestone powder content results a better gel formation that leads to a denser microstructure. This is the result of the reduction of water absorption when limestone powder content increases.

On the other hand, increasing the concentration of NaOH leads to high degree of geopolymerization (Sakonwan et al. 2014) this produce a more denser gel that minimize the amount of absorbed water by the samples.

Table 4 Effect of limestone replacement and NaOH concentration variation on water absorption of fine soil based mortar

Mixes	Water absorption%		
	10M	12M	14M
S100L0	14.75	13.33	12.37
S75L25	14.32	13.36	11.64
S50L50	14.41	13.44	10.67
S25L75	13.32	11.75	9.69
S0L100	10.50	9.08	6.83

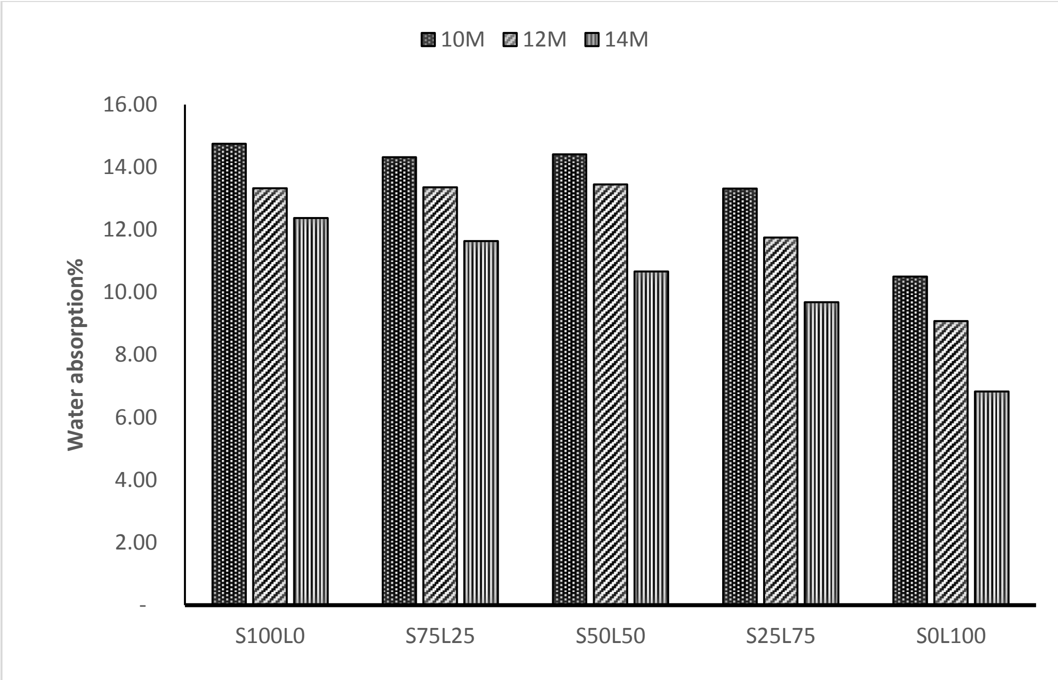
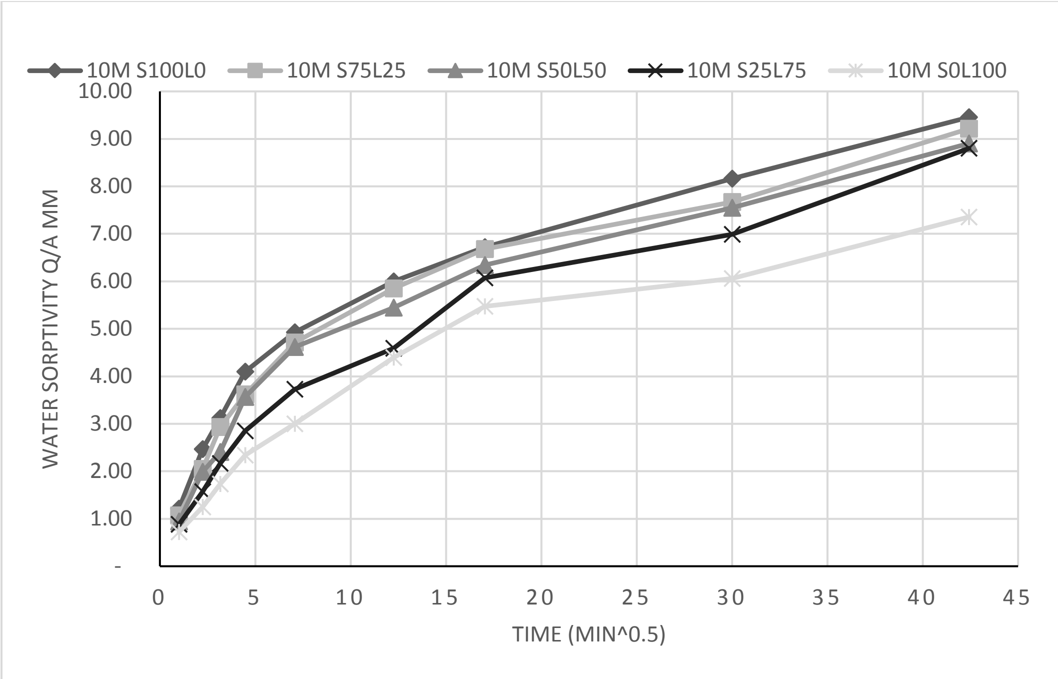


Fig. 6. Effect of limestone replacement and NaOH concentration variation on water absorption of fine soil based mortar

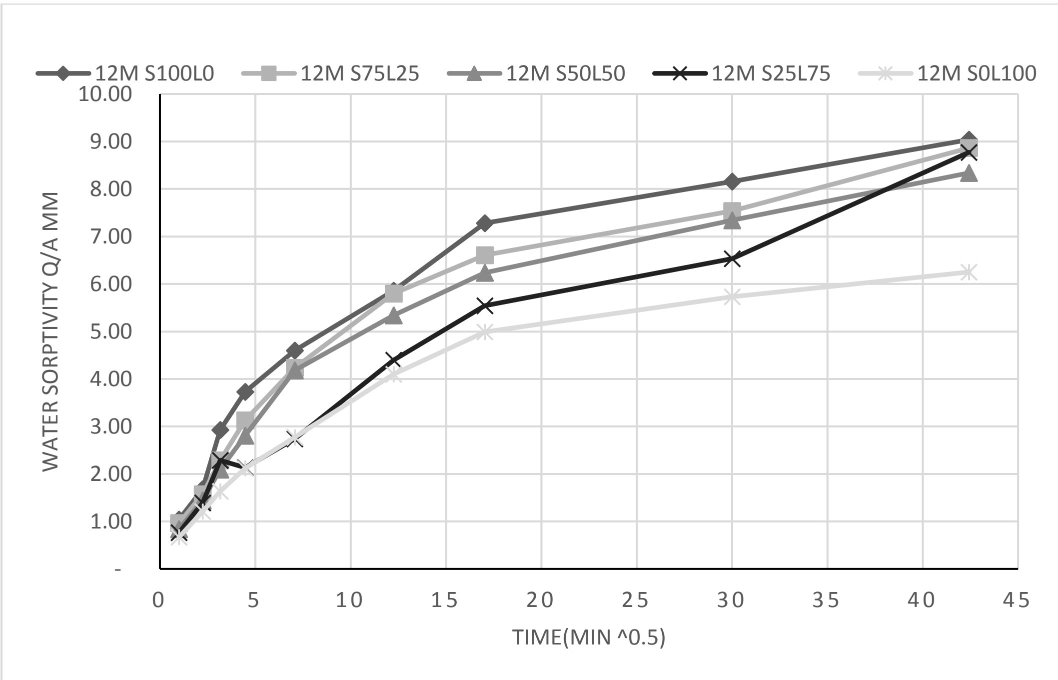
Water Sorptivity:

The results of the water sorptivity are shown in the figure 6. a, b and c. Increasing limestone powder content results a better gel formation that leads to a more dense microstructure. This is the result of the reduction of water absorption when limestone powder content increases.

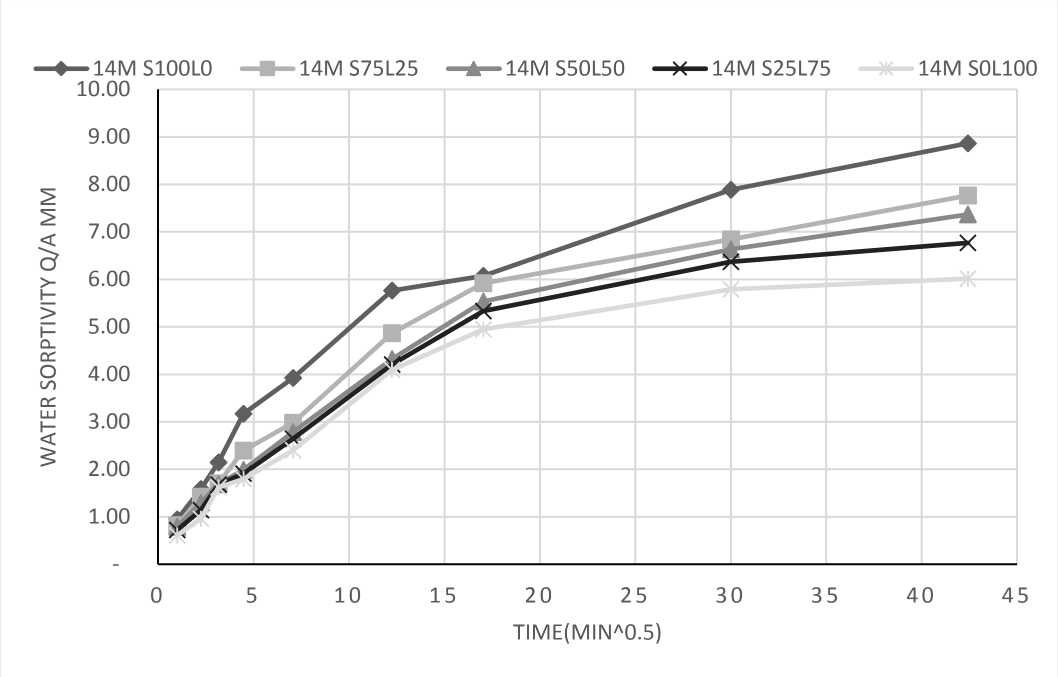
On the other hand, increasing the concentration of NaOH leads to high degree of geopolymerization (Sakonwan et al. 2014) this produce a more denser gel that minimize the amount of suction water by capillary action.



a) Effect of limestone replacement and NaOH (M10) concentration variation on sorptivity of fine soil based mortar



b)Effect of limestone replacement and NaOH (M12) concentration variation on sorptivity of fine soil based mortar

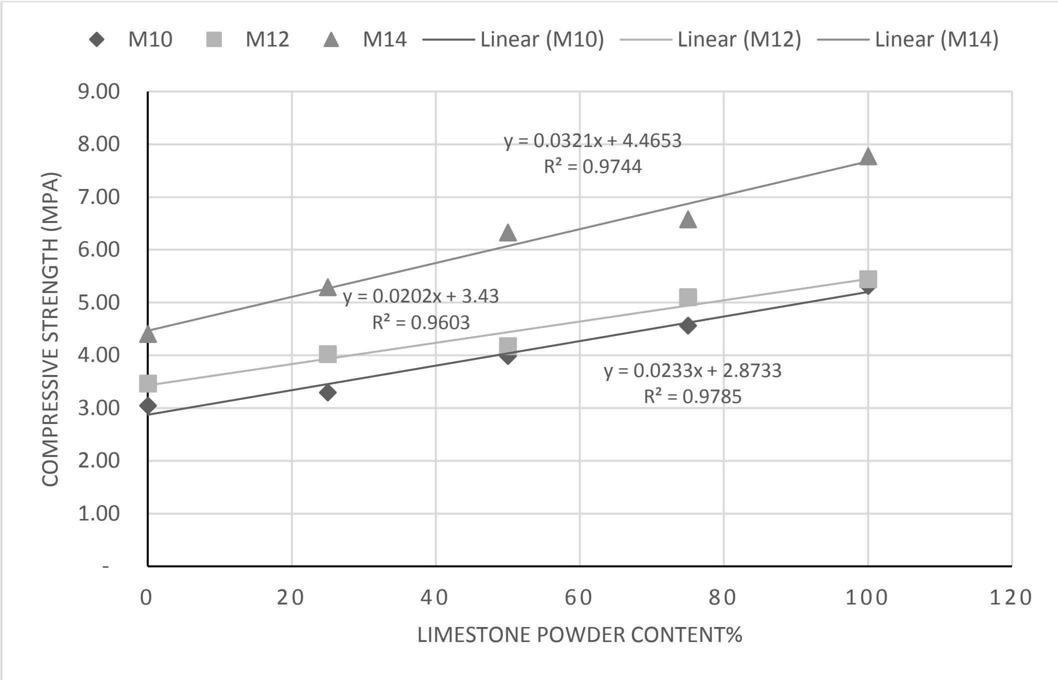


c)Effect of limestone replacement and NaOH (M14) concentration variation on sorptivity of fine soil based mortar

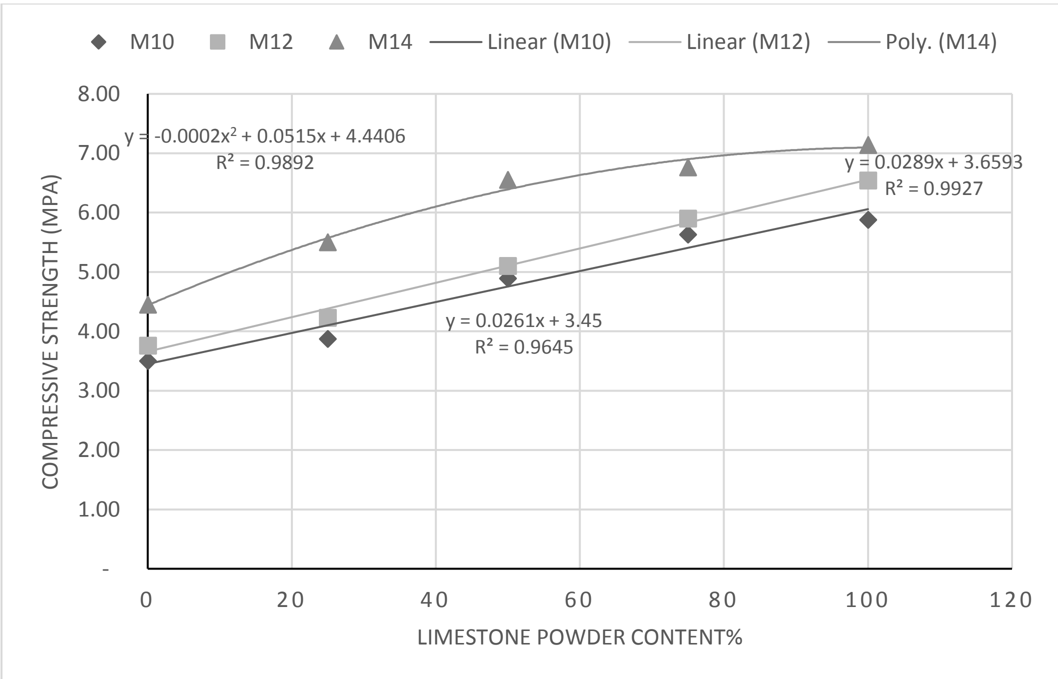
Fig. 7. Effect of limestone replacement and NaOH concentration variation on sorptivity of fine soil based mortar

Correlation between hardened properties:

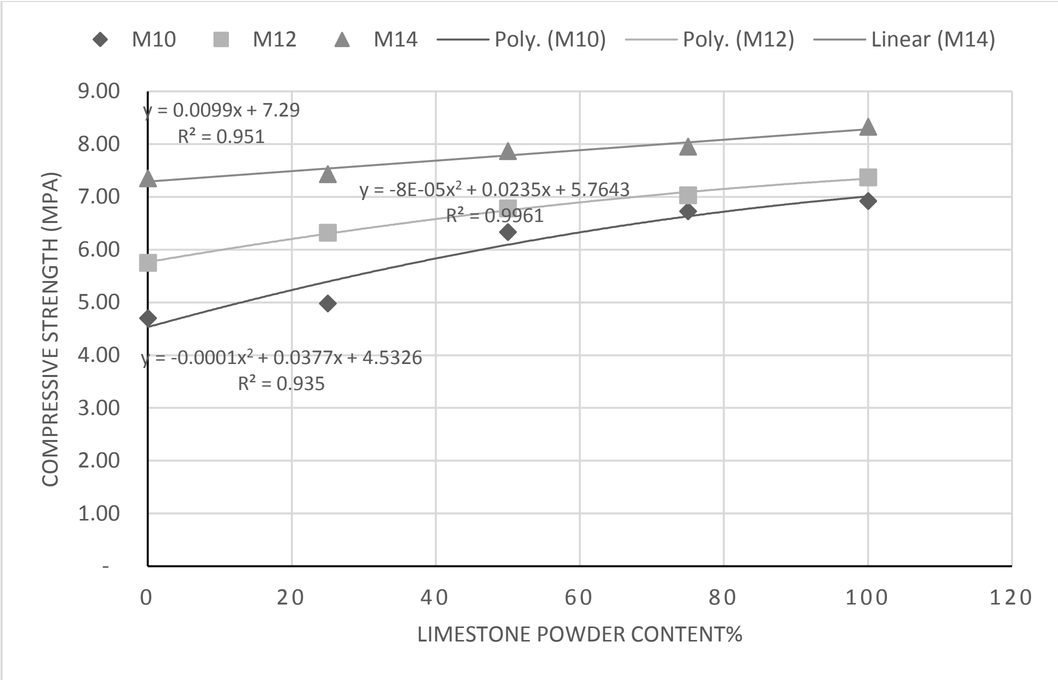
After obtaining the results, it is significant to determine the relationship between the properties. There are several factors affect the mechanical properties of concrete like w/c, aggregate. ect. As stated earlier, most of the mechanical properties of concrete related to compressive strength of concrete. In this study, the effect of limestone powder replacement and variation of NaOH concentration were investigated. Simultaneously the correlating and relationship between the achieved results was also studied. There are close relationship between limestone powder replacement and compressive strength (R^2:0.98), (R^2:0.96) and (R^2: 0.97) at age of 14, (R^2:0.96), (R^2:0.99) and (R^2: 0.99) at age of 21, (R^2:0.93), (R^2:0.99) and (R^2: 0.95) at age of 28 for NaOH concentrations 10M, 12M and 14M respectively. At the same time, there is a close relationship between limestone powder replacement and water absorption (R^2:0.96), (R^2:0.98) and (R^2: 0.98) at age of 28 for NaOH concentrations 10M, 12M and 14M respectively. Furthermore, there is a close relationship between limestone powder replacement and water sorptivity (R^2:0.98), (R^2:0.86) and (R^2: 0.94) at 50minutes at age of 28 for NaOH concentrations 10M, 12M and 14M respectively. From the R^2 values, it can be concluded that there is an excellent correlation between the limestone powder replacement ratio and mechanical and durability properties even at the age of 28 days as shown in Fig. 8.



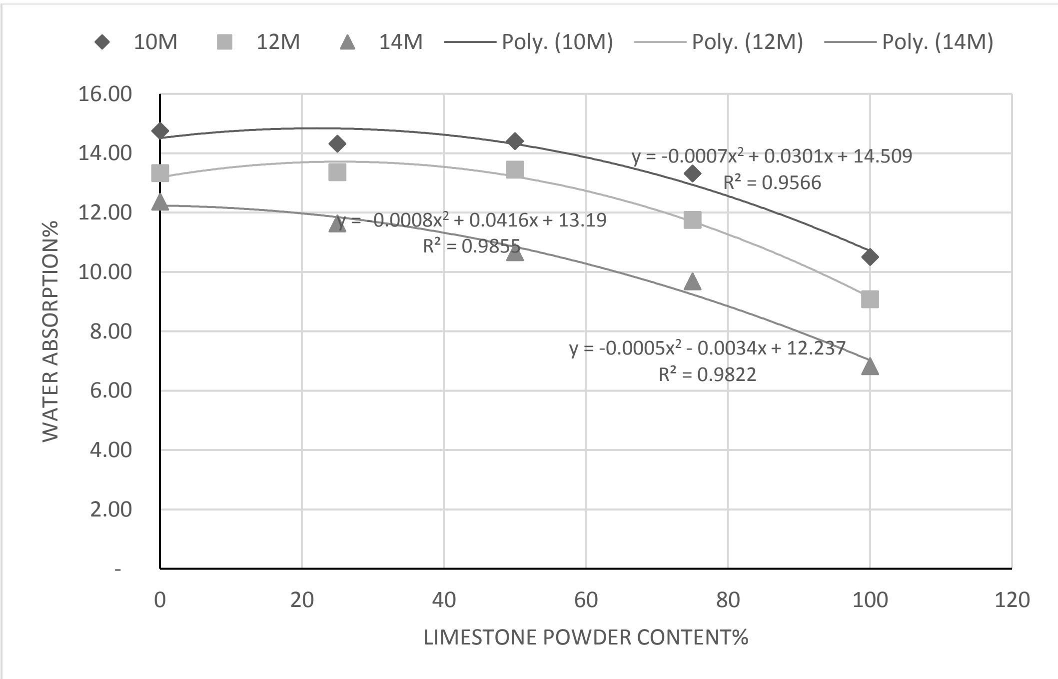
a) Replacement ratio of limestone versus compressive strength at 14 day



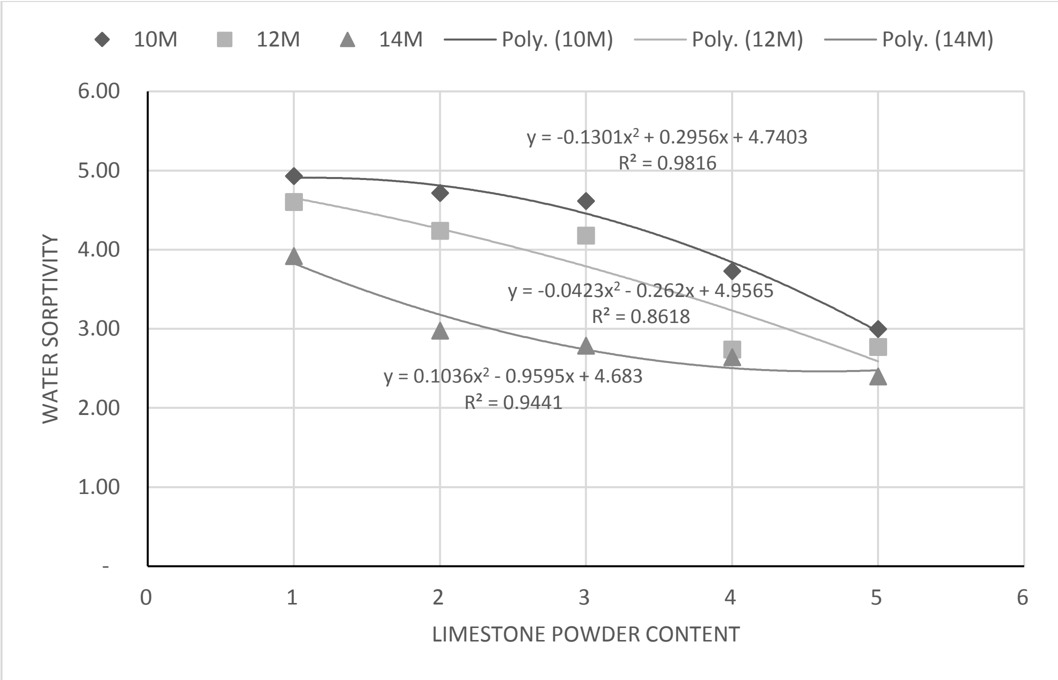
b) Replacement ratio of limestone versus compressive strength at 21 day



c) Replacement ratio of limestone versus compressive strength at 28 day



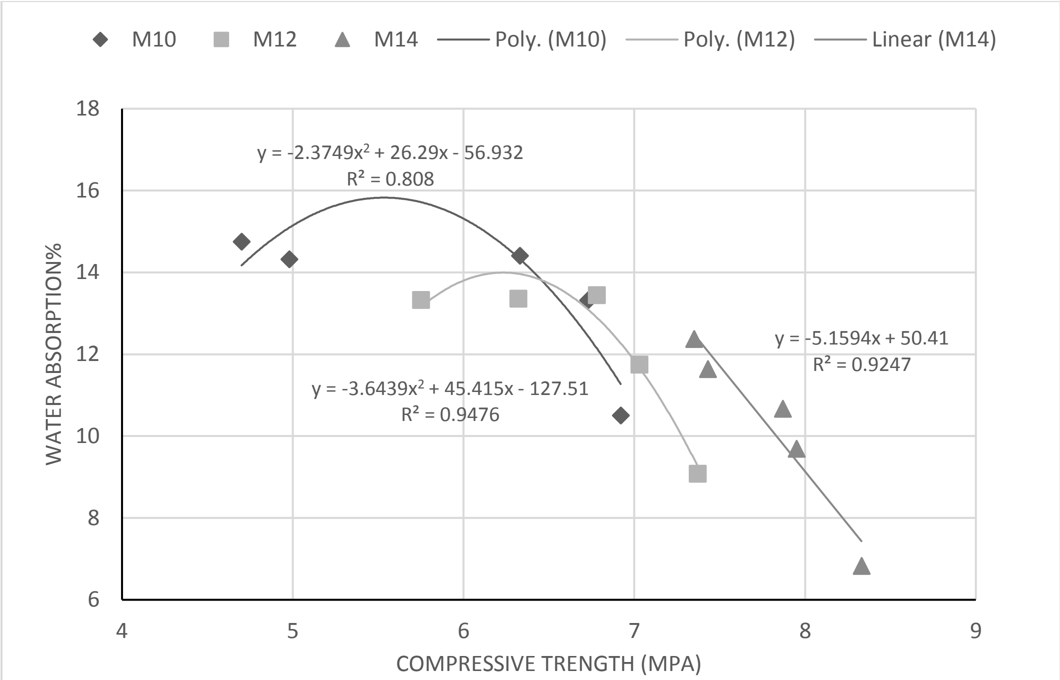
d) Replacement ratio of limestone versus water absorption



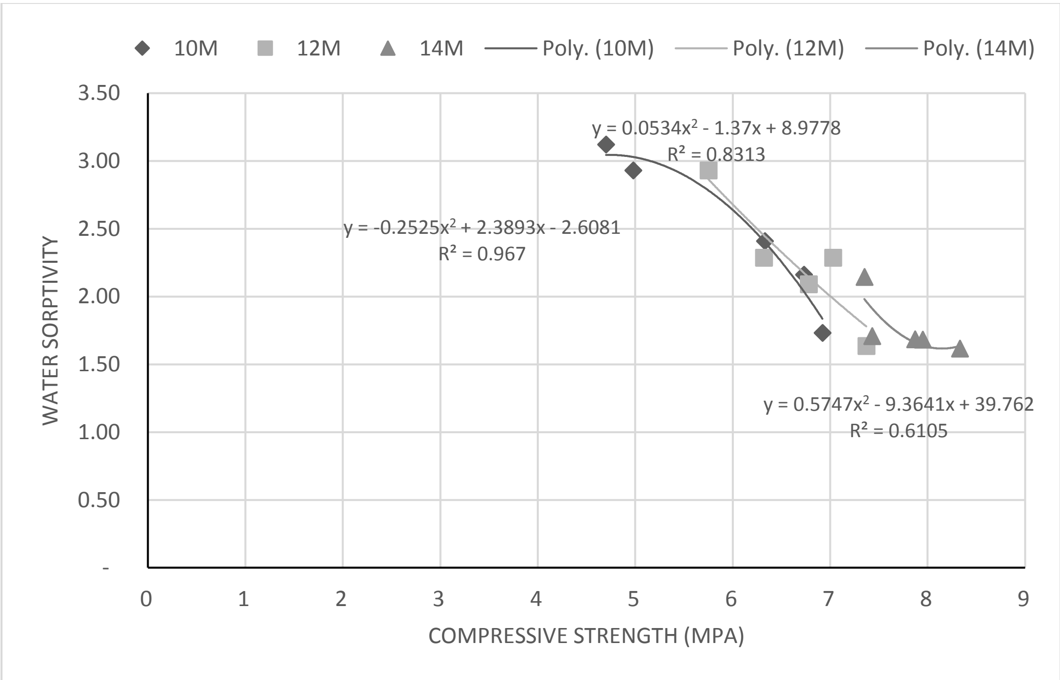
d) Replacement ratio of limestone versus sorptivity (50 min)

Figure. 8**.** Effect of limestone replacement ratio on the properties of alkali activated mortar.

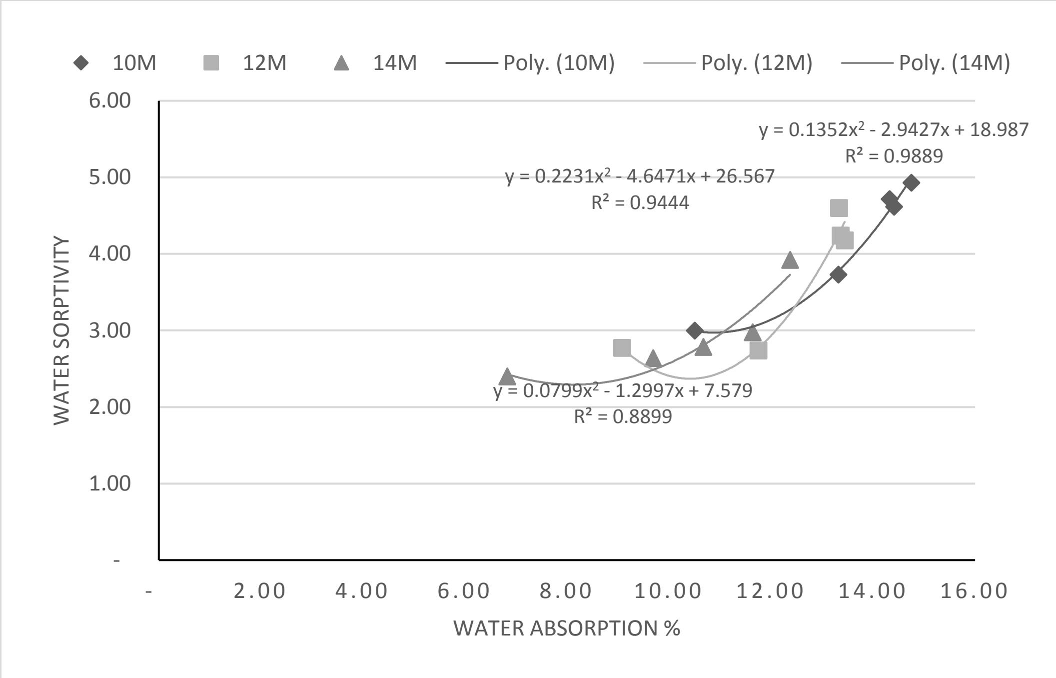
On the other hand, the results also shown noticeable close relationships between mechanical and durability properties of alkali activated mortar as illustrated in Fig. 9. The good correlation exhibits high coefficient (R^2) values which indicate that the mechanical and durability properties of alkali activated mortar improved and deteriorated with similar factors or effects. Moreover, good relationships presence between the hardened performances of alkali activated mortar and the concentration of alkali solution (NaOH). The correlation between hardened performance and concentration of alkali activated exhibits high coefficient (R^2) value as shown in Fig. 9. Generally, it can be deduced that there are excellent relationships between hardened properties and concentration of alkali activated mortar despite the incorporation of limestone powder. Limestone powder replacement had significant effect on the hardened properties. Therefore, in order to achieve superior alkali activated specimens exhibit good mechanical properties, good durability properties are inevitable requirement.



a) Water absorption vs compressive strength



b)Water sorptivity (10min) vs compressive strength



c)Water sorptivity (50min)Vs Water absorption

Figure 9 Correlation between the hardened properties of alkali activated mortar

Statistical analysis:

An analysis of variance model with a significant level of 0.05 is conducted to evaluate the variation of the alkali activated mortar performance with different level of limestone powder replacement ratio and/or different alkali solution concentration in a quantitative form. For this, compressive strength, water absorption and sorptivity of the mortar were designated as the dependent variables while the concentration of alkali activated solution and replacement level of limestone powder were the independent factors. In order to determine significant factors with a p-level of smaller than 0.05, a statistical analysis was carried out. The P-level that is smaller than 0.05 showed that the related parameter is a significant parameter in the resulting performance. In addition, percent contribution was also determined to find out the degree of effectiveness of the parameter on the resulting performance. If this value is higher, then it can be accepted as the influence of the parameter is significant on the resulting performance. Meanwhile, the mechanical and durability performances of the specimens significantly improved with the replacement of limestone powder and the best concentration was 14M.

Table 5 Statistical evaluation of the test result

Dependent variable	Independent variable	Sequential sum of square	Mean Square	Computed F	P-Value	Significance	Contribution %
Compressive strength	LSP replacement	6,115.41	6115.41	136.31	0.001	Yes	97.59
	NaOH	7.97	7.97	340.41	0.034	Yes	0.13
	Curing time	97.9938	97.9938	15696.33	0.005	Yes	1.56
	Error	44.88					0.72
	Total	6,266.26					
water absorption	LSP replacement	5,702.90	5702.9	31.27	0.011	Yes	86.34
	NaOH	7.95	7.95	164.64	0.05	Yes	0.12
	Error	894.40					13.54
	Total	6,605.25					
Water Sorptivity	LSP replacement	6,146.80	6146.77	178.63	0.001	Yes	98.22
	NaOH	7.99	7.99	1136.53	0.019	Yes	0.13
	Error	103.21					1.65
	Total	6,258.00					

Conclusion:

On the basis of the results achieved these points can be drawn.

1 Increasing limestone powder content in the mixes leads to increase the compressive strength. At the same time increasing NaOH concentration leads to increase compressive strength.

2 Increasing limestone powder content in the mixes leads to decrease water absorption. At the same time increasing NaOH concentration leads to decrease water absorption.

3 Increasing limestone powder content in the mixes leads to decrease water sorptivity. At the same time increasing NaOH concentration leads to decrease water sorptivity.

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