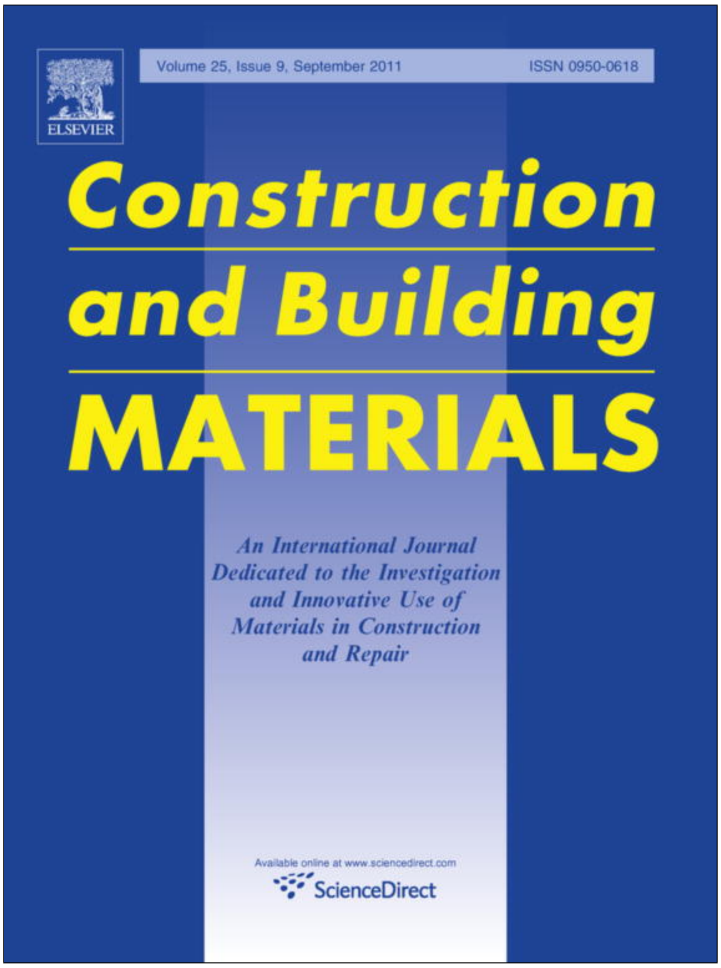
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Composition, strength and workability of alkali-activated metakaolin based mortars

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ABSTRACT

This study has investigated the joint effect of several factors on the workability and mechanical strength of alkali-activated metakaolin based mortars. The factors analysed through a laboratory experiment of 432 specimens, pertaining to 48 different mortar mixes were, sodium hydroxide concentration (10 M, 12 M, 14 M, 16 M), the superplasticizer content (1%, 2%, 3%) and the percentage substitution of metakaolin by calcium hydroxide in the mixture (5%, 10%). The results show that the workability decreases with the concentration of sodium hydroxide and increases with the amount of calcium hydroxide and superplasticizer. The results also show that the use of 3% of superplasticizer, combined with a calcium hydroxide content of 10%, allows improving the mortar flow from less than 50% to over 90%, while maintaining a high compressive and flexural strength.

1. Introduction

To make Portland cement clinker limestone is heated with a source of silica in a kiln at temperatures well over 1350 °C. The production of one tonne of cement generates 0.55 tonnes of chemical CO_2 and requires an additional 0.39 tonnes of CO_2 in fuel emissions for baking and grinding, accounting for a total of 0.94 tonnes of CO_2 [1]. Other authors [2] report that the Portland cement industry emitted in 2000, on average, 0.87 kg of CO_2 for every kg of cement produced. Current estimates of world cement manufacture are near 3000 Mt (million tonnes)/year. Although Portland cement demand is decreasing in industrial nations, it is increasing dramatically in developing countries. Global demand will have increased almost 200% by 2050 from 2010 levels (Fig. 1). This is particularly serious in the current context of climate change caused by carbon dioxide emissions worldwide, causing a rise in sea level and the occurrence of natural disasters and being responsible for a future meltdown in the world economy [4,5]. Furthermore, the search for more durable binders relates to the fact that reinforced Portland cement concrete structures deterioration is a very common phenomenon. Beyond the durability problems originated by imperfect concrete placement and curing operations, the real issue about Portland cement durability is related to the intrinsic properties of that material. It presents a higher permeability that allows water and other aggressive elements to enter, leading to carbonation and chloride ion attack resulting in corrosion problems. This implies expensive conservation actions or building new structures. Research works carried out so far in developing alkali-activated binders, show that this new material is likely to have enormous potential to become an alternative to Portland cement. Alkali-activated concrete have been receiving increased attention, due to the need of reducing green house gas emissions generated by Portland cement and to the need of new binders with enhanced durability performance [6–8]. Although research in this field has been published as "alkali-activated" binders, the term "geopolymer" is the generally accepted name for this technology. Geopolymerisation involves a chemical reaction between various alumino-silicate oxides with silicates under highly alkaline conditions, yielding polymeric Si–O–Al–O bonds indicating that any Si–Al materials could become sources of geopolymerisation. Alkali-activated binders generates 80% less carbon dioxide than Portland cement [9]. Weil et al. [10] mentioned that in comparison to Portland cement concrete the global warming potential (GWP) of alkali-activated concrete is 70% lower. The high cost of alkali-activated binders is one of the major factors which still remains a severe disadvantage over Portland cement [11–13]. Currently alkali-activated binders only becomes economic competitive for high performance structural purposes. However, future increase cost of Portland cement due to European Emissions Trading Scheme (ETS) that will put a price on carbon dioxide emissions generated during clinker production will reduce the economic advantage of this material. In the short term the above cited disadvantage means that the study of alkali-activated applications should focus on high cost materials such as, commercial concrete repair mortars. Pacheco-Torgal et al. [14–16] show that alkali-activated mortars can be as much as seven times cheaper than current commercial repair mortars. But if

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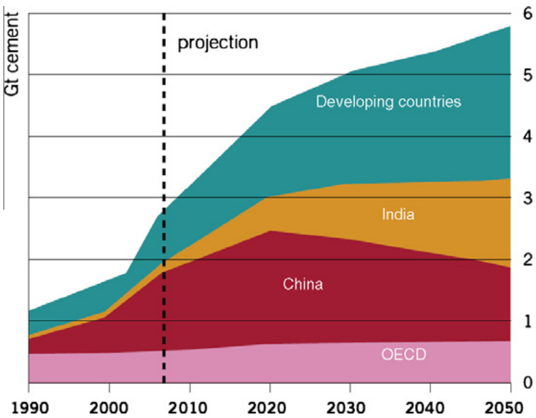

Fig. 1. Portland cement demand [3].

Table 1
Chemical composition of metakaolin.

SiO_2	Al_2O_3	Fe_2O_3	K_2O	Na_2O	MgO	TiO_2	Other minor oxides
50.75	43.48	2.45	–	0.04	0.11	0.57	2.6

the cost to bond strength ratio were compared the differences are even higher, with the cost of the cheapest commercial repair mortar being 13.8 times higher than the alkali-activated mortars. Alkali-activated mortars and concretes present a stiff workability behaviour arising from the use of viscous compounds such has sodium silicate and sodium hydroxide. Several authors have reported placement difficulties related to the low workability of alkali-activated mortars. Some authors [17] show that several superplasticizers used in the Portland cement concrete industry lost their fluidifying properties for alkali-activated mortars. Other authors [18] found out that the use of a superplasticizer leads to an improvement of alkali-activated mortars workability but they can also contribute to a reduction on compressive strength depending on the sodium silicate to NaOH ratio. Sathonsaowaphak et al. [19] reported that workable ranges of sodium silicate/NaOH ratios and NaOH concentration are between 0.67–1.5 and 7.5–12.5 M. Also Rangan [20] confirmed that the addition of a naphthalene sulfonate-based superplasticizer improves the workability of fly ash geopolymer mixtures, however, a superplasticizer content above 2% is responsible for a slight degradation of compressive strength. Therefore, the purpose of this paper is to understand how the composition of alkali-activated mortars influences its workability and its mechanical strength.

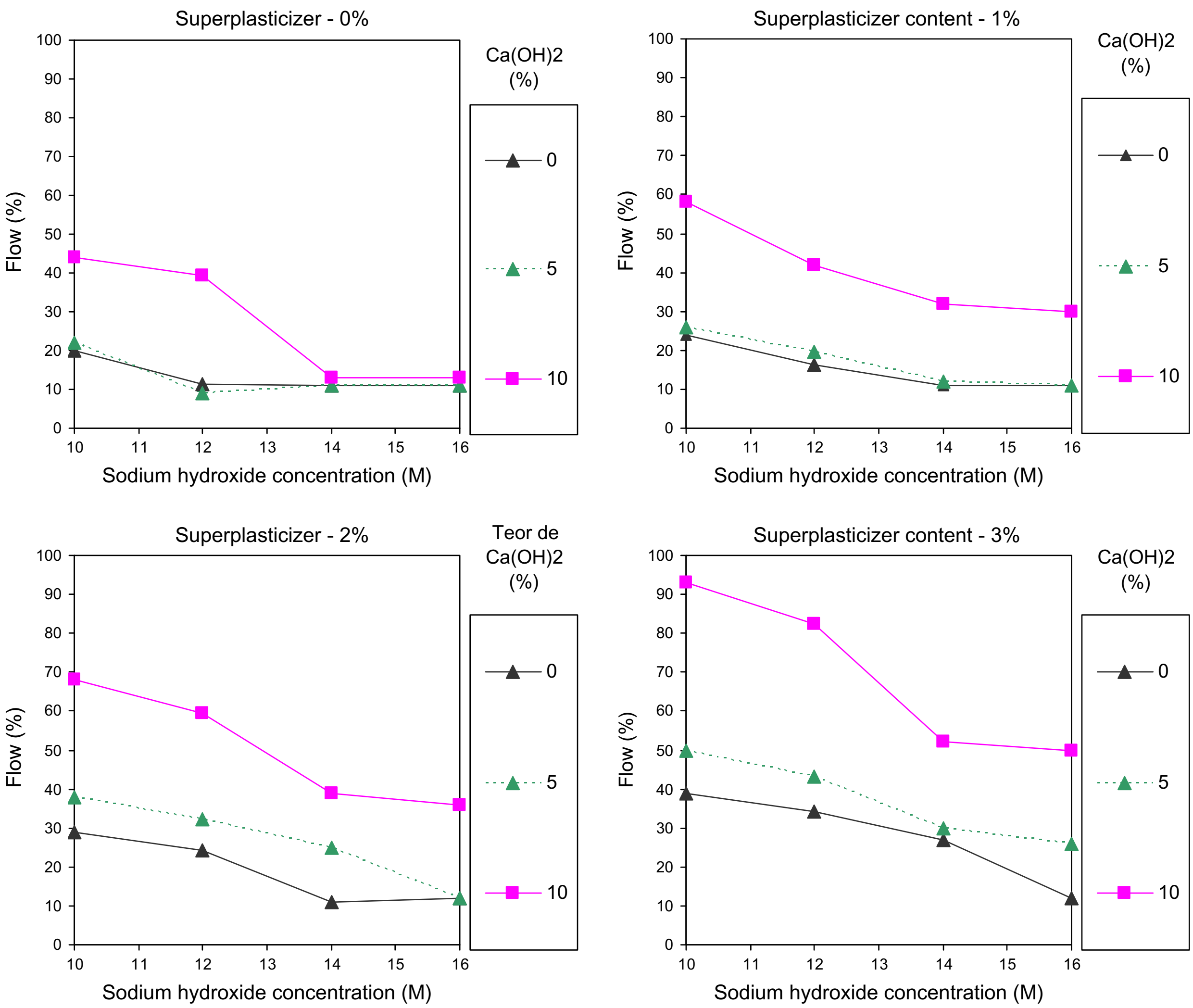

Fig. 2. Flow versus sodium hydroxide concentration for several contents of superplasticizer and calcium hydroxide.

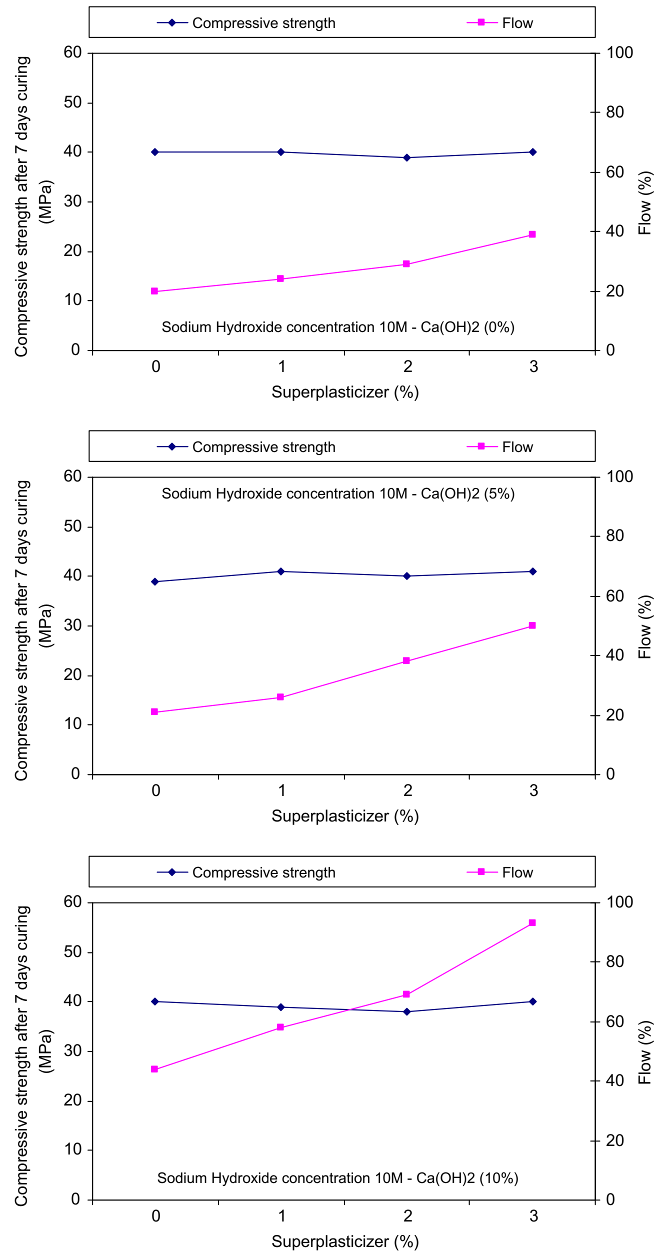

Fig. 3. Compressive strength for 7 days curing versus flow in mortars with a 10 M sodium hydroxide concentration and a calcium hydroxide percentage of 10% for several contents of superplasticizer.

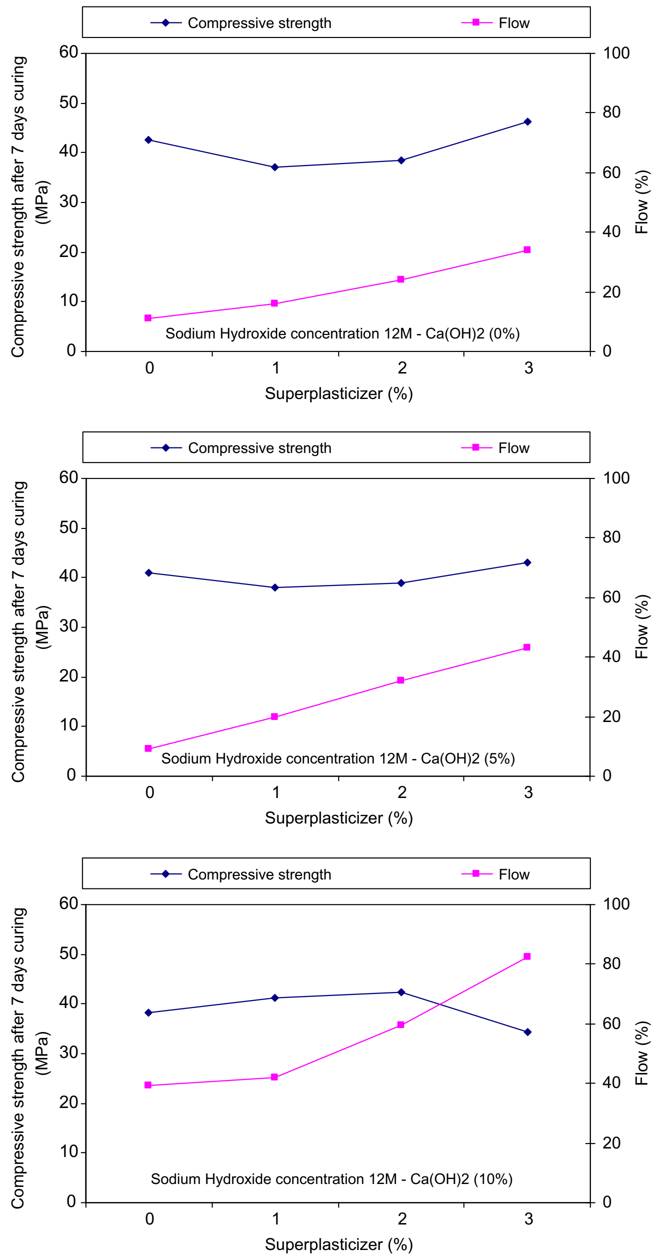

Fig. 4. Compressive strength for 7 days curing versus flow in mortars with a 12 M sodium hydroxide concentration and a calcium hydroxide percentage of 10% for several contents of superplasticizer.

2. Experimental work

2.1. Materials

The metakaolin used in this study was subjected to a thermal treatment at 650 °C during a few seconds using a flash calcination apparatus. Its chemical composition is shown in Table 1. The fine aggregate used was crushed sand from the same mine with a specific gravity of 3.0 a 24 h water absorption of 1.0%, and a finesse modulus of 2.8. The superplasticizer has been provided by MAISOL – FPR, with a density of 1.100 ± 0.005 g/cm^3. A hydrated commercial lime powder supplied by Lusical with more than 70% of CaO and a density of 0.46 g/cm^3 was also used.

2.2. Mixture proportions and synthesis

The factors considered in this investigation led to the manufacture of 48 different mixes (432 specimens). The factors analysed were, sodium hydroxide concentration (10 M, 12 M, 14 M and 16 M), the superplasticizer content (1%, 2% and 3%) and the percentage substitution of metakaolin by calcium hydroxide in the mixture (5% and 10%). The mass ratio of sand/metakaolin/activator used was 2.2/1/1. Previous trials showed that a higher sand content leads to a very stiff behaviour and a lower one leads to liquid like mortar. The alkaline activator was prepared prior to use. An activator with sodium hydroxide and sodium silicate solution (Na_2O = 13.5%, SiO_2 = 58.7%, and water = 45.2%) was used with a mass ratio of 1:2.5. Previous investigations showed that this ratio lead to the highest compressive strength results in alkali-activated mortars. Distilled water was used to dissolve the sodium hydroxide flakes to avoid the effect of unknown contaminants in the mixing water. The alkaline activator was prepared prior to use. Alkali-activated mortars were a mixture of aggregates, metakaolin, calcium hydroxide and alkaline silicate solution. The sand, metakaolin and calcium hydroxide were dry mixed before added to the activator. No extra water has been added.

2.3. Workability

The workability assessment has been conducted with a truncated conical mould and a jolting table according to the EN 1015-3.

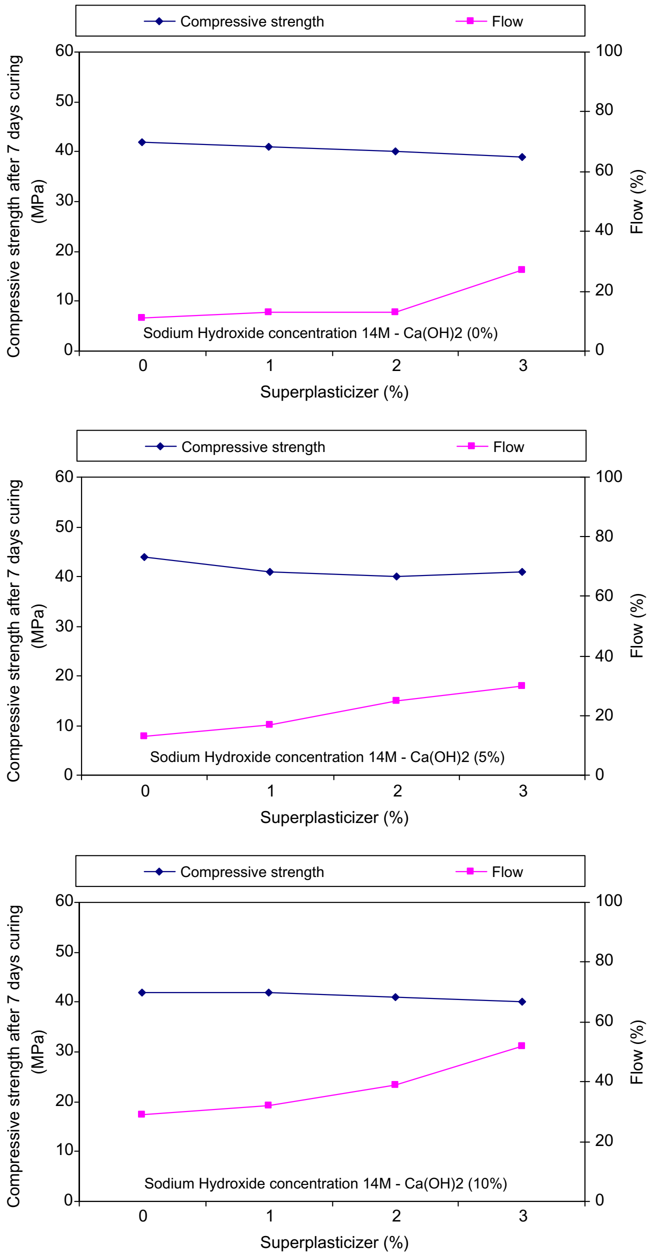

Fig. 5. Compressive strength for 7 days curing versus flow in mortars with a 14 M sodium hydroxide concentration and a calcium hydroxide percentage of 10% for several contents of superplasticizer.

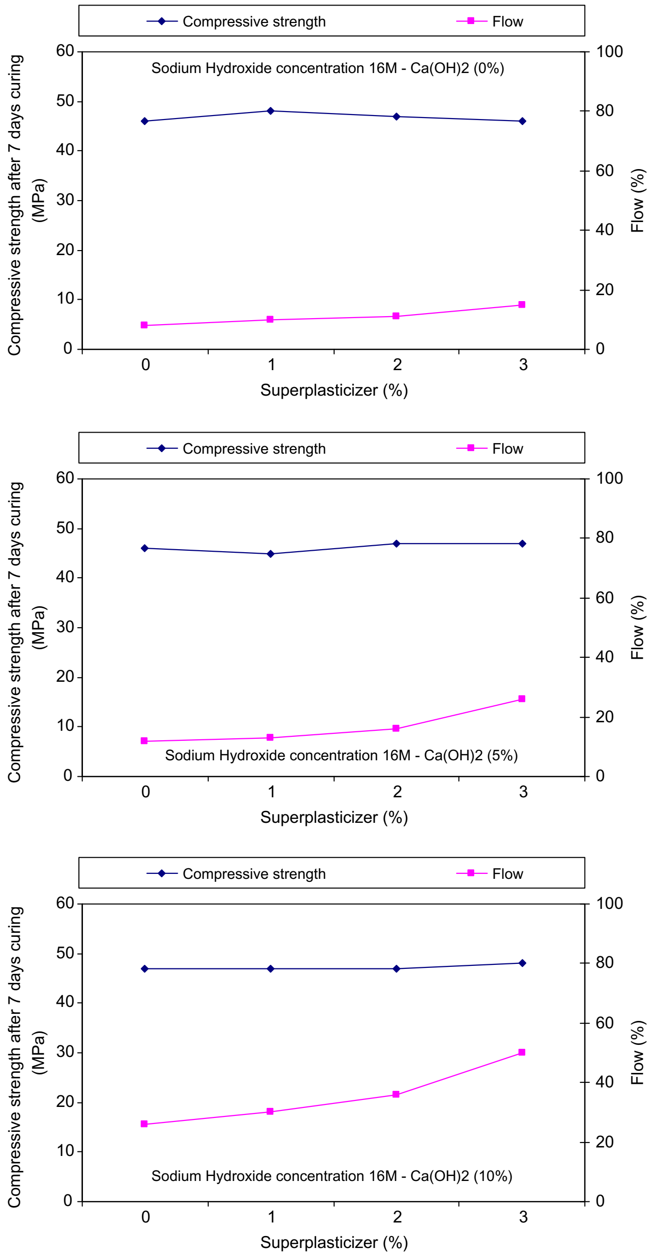

Fig. 6. Compressive strength for 7 days curing versus flow in mortars with a 16 M sodium hydroxide concentration and a calcium hydroxide percentage of 10% for several contents of superplasticizer.

2.4. Compressive and flexural strength testing

Compressive and flexural strength data was obtained using 160 × 40 × 40 mm^3 cubic specimens according to EN 1015-11. The fresh mortar were cast and allowed to set at room temperature for 24 h before being removed from the moulds and kept at room temperature (20 °C) until tested in compression and flexural strength. Compressive strength for each mortar mixture was obtained from an average of 3 specimens from those broken in flexure.

3. Results and discussion

Fig. 2 presents the results of the alkali-activated mortars flow according to the sodium hydroxide concentration for several contents of superplasticizer and calcium hydroxide. The results show that alkali-activated mortars without superplasticizer show a flow below 50%. Mortars with increase superplasticizer content show an increase flow. The mortars containing a high calcium hydroxide show a high flow because this mixture has a low percentage of metakaolin. Since metakaolin has a high Blaine fineness it needs a high liquid phase in order to be dissolved. Flow is also reduced with high sodium hydroxide concentration. The highest flow was achieved by mortars with a sodium hydroxide concentration of 10 M and a calcium hydroxide of 10% (Fig. 3d). The use of a superplasticizer content of 3% combined with a calcium hydroxide content of 10%, allows increasing a mortar flow of less than 50% to over 90%. Similar findings were recently reported [19]. Figs. 3–6 show the compressive strength for 7 days curing versus flow in alkali-activated mortars according to the sodium hydroxide concentration and several contents of superplasticizer and calcium hydroxide. In the mortars with a sodium hydroxide concentration of 10 M, the flow increase due to the increase in the superplasticizer content is associated to an almost constant strength level, which is a different behaviour from the one reported by other authors

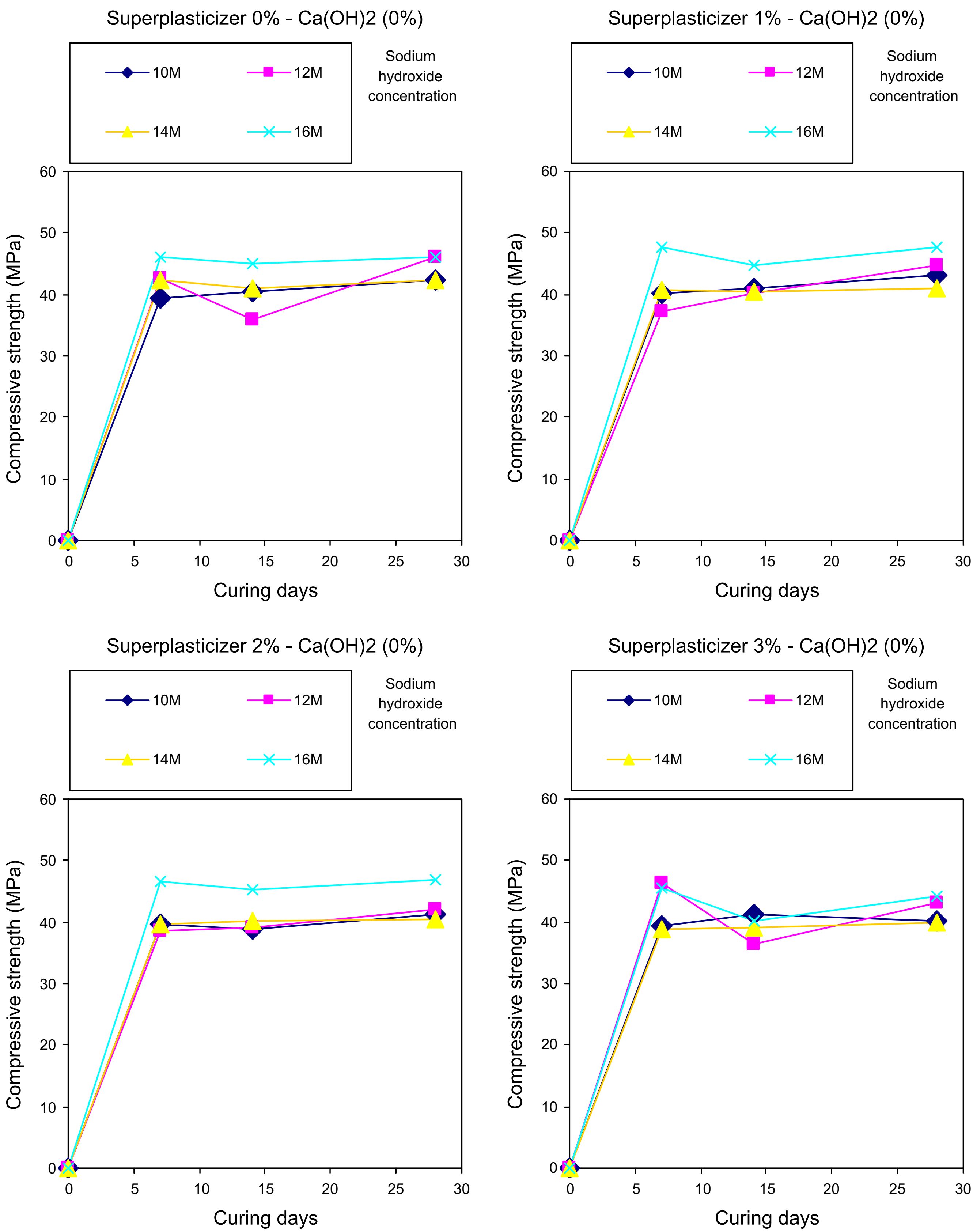

Fig. 7. Compressive strength according to age for mortar mixtures with 0% of calcium hydroxide and several sodium hydroxide concentrations and superplasticizer contents.

[16,17]. As for the mortars with a sodium hydroxide concentration of 12 M and a 10% calcium hydroxide, when the superplasticizer content increase from 2% to 3% a flow increase is observed but at the same time a compressive reduction takes place. This confirms

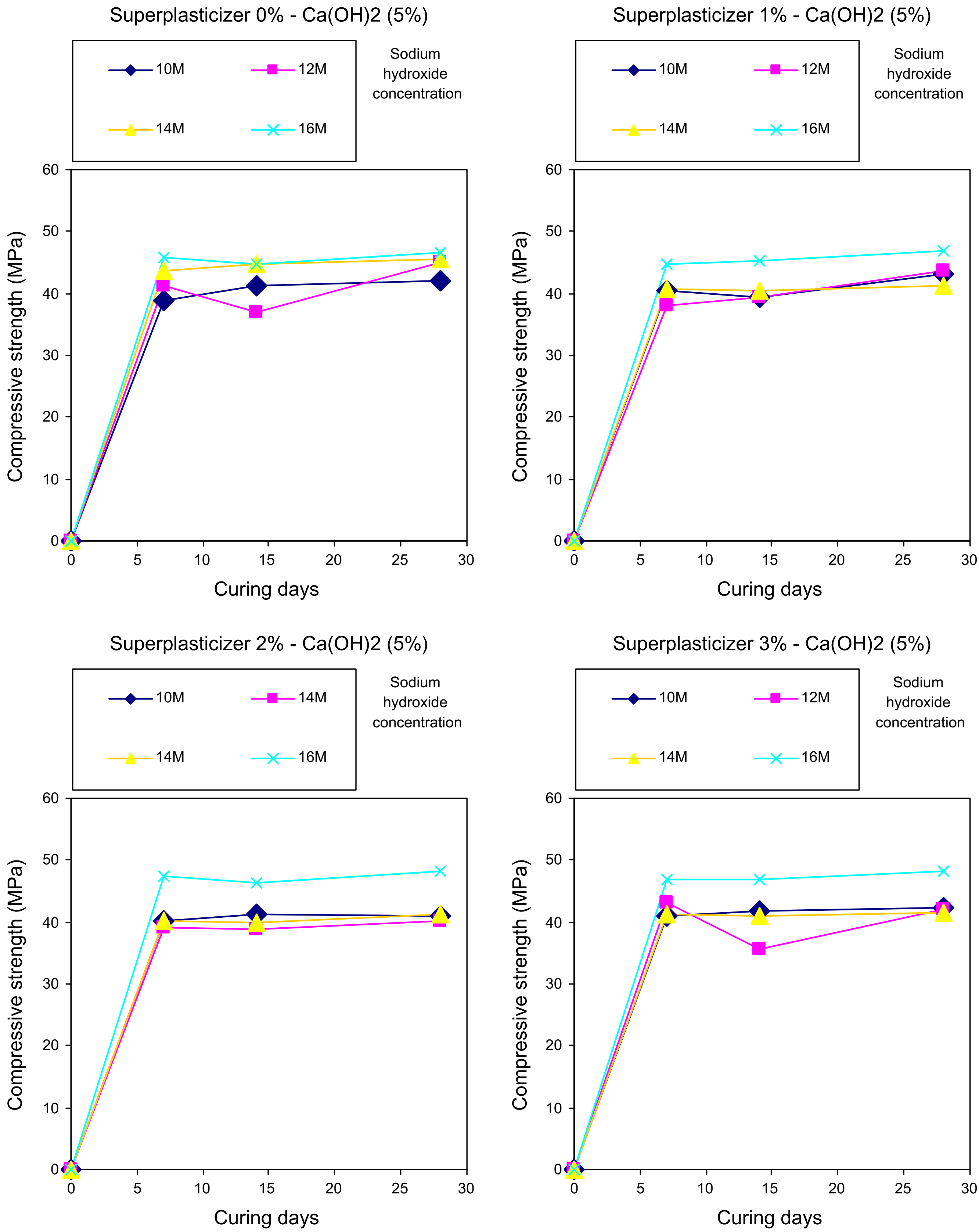

Fig. 8. Compressive strength according to age for mortar mixtures with 5% of calcium hydroxide and several sodium hydroxide concentrations and superplasticizer contents.

previous findings [17,20] and could be explained by high air entrained content. The compressive strength of alkali-activated mortars according to the curing days is showed in Figs. 7–9. The results show that higher sodium hydroxide concentrations lead to a com-

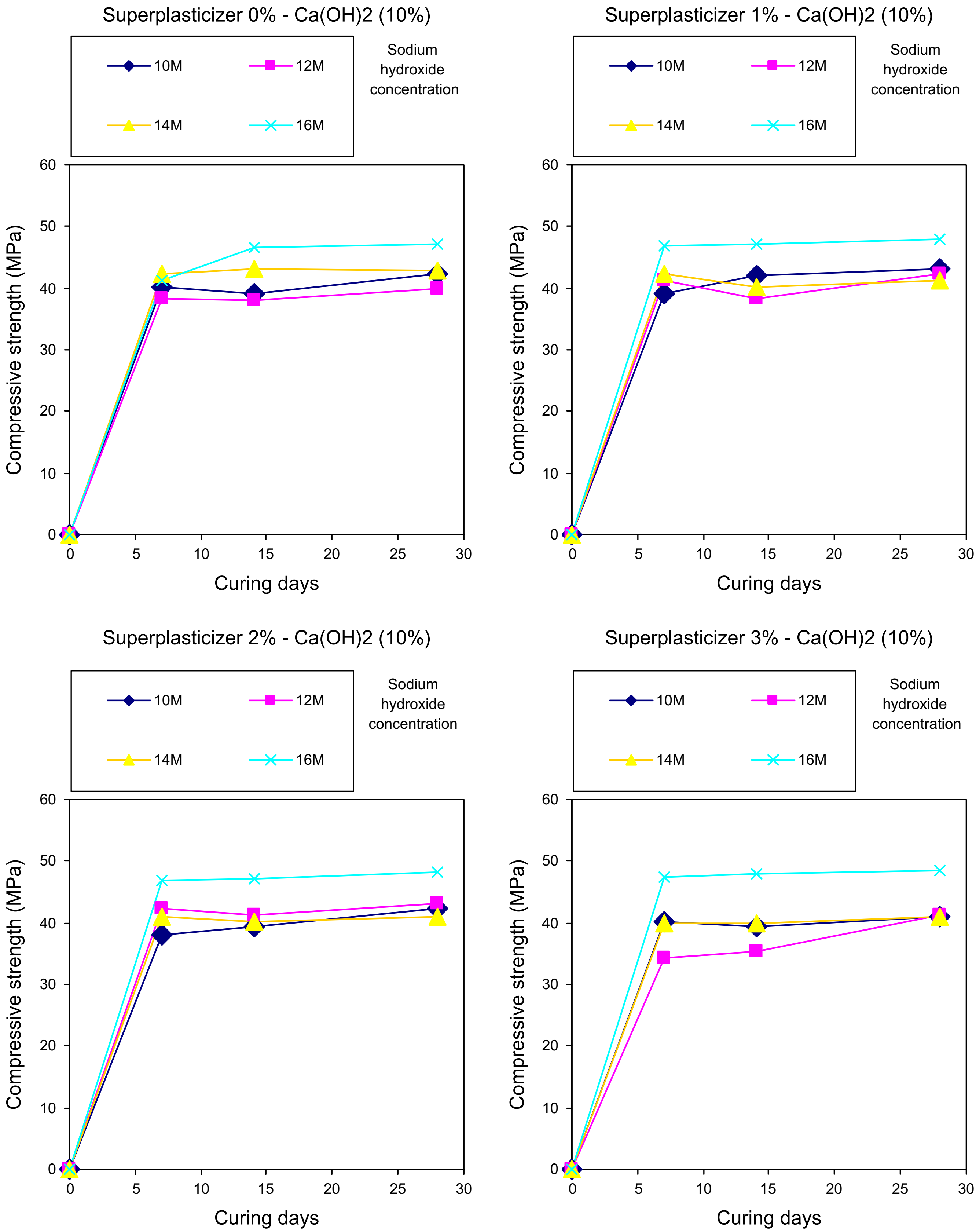

Fig. 9. Compressive strength according to age for mortar mixtures with 10% of calcium hydroxide and several sodium hydroxide concentrations and superplasticizer contents.

pressive strength increase, but that only happens beyond 7 days curing. Higher concentrations of alkaline solution raises the pH which increases the dissolution and solubility of the aluminosilicate mineral waste and provides positive ions to balance the neg-

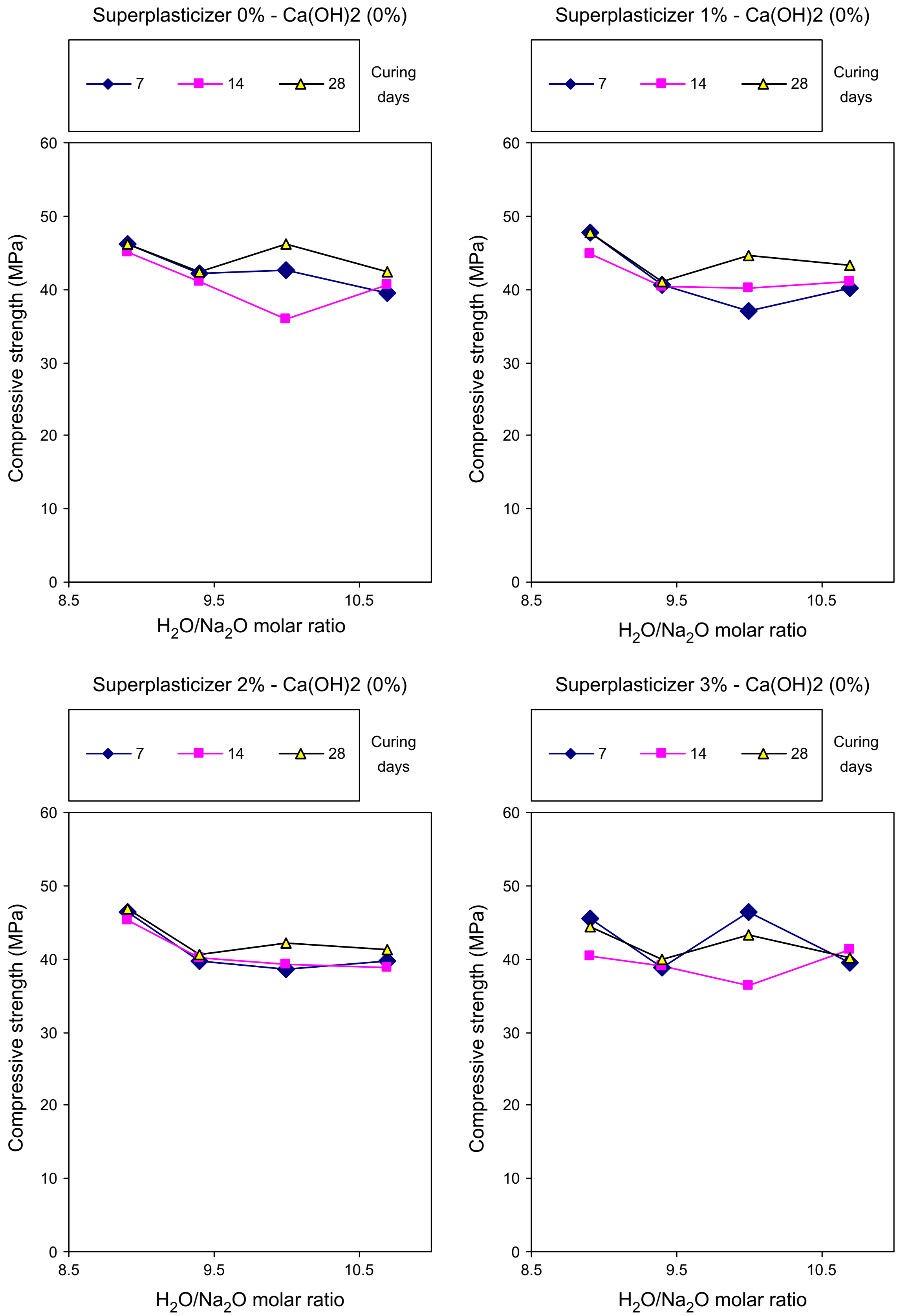

Fig. 10. Compressive strength versus H_2O/Na_2O atomic ratio for mortars with 0% of calcium hydroxide and several superplasticizer contents.

ative charge of the aluminate group [21,22]. The adverse effect reported by Lee et al. [23] related to reduction in strength due to excess of alkali have not been confirm. With the exception of the mix with a sodium hydroxide concentration of 12 M and a 3% of

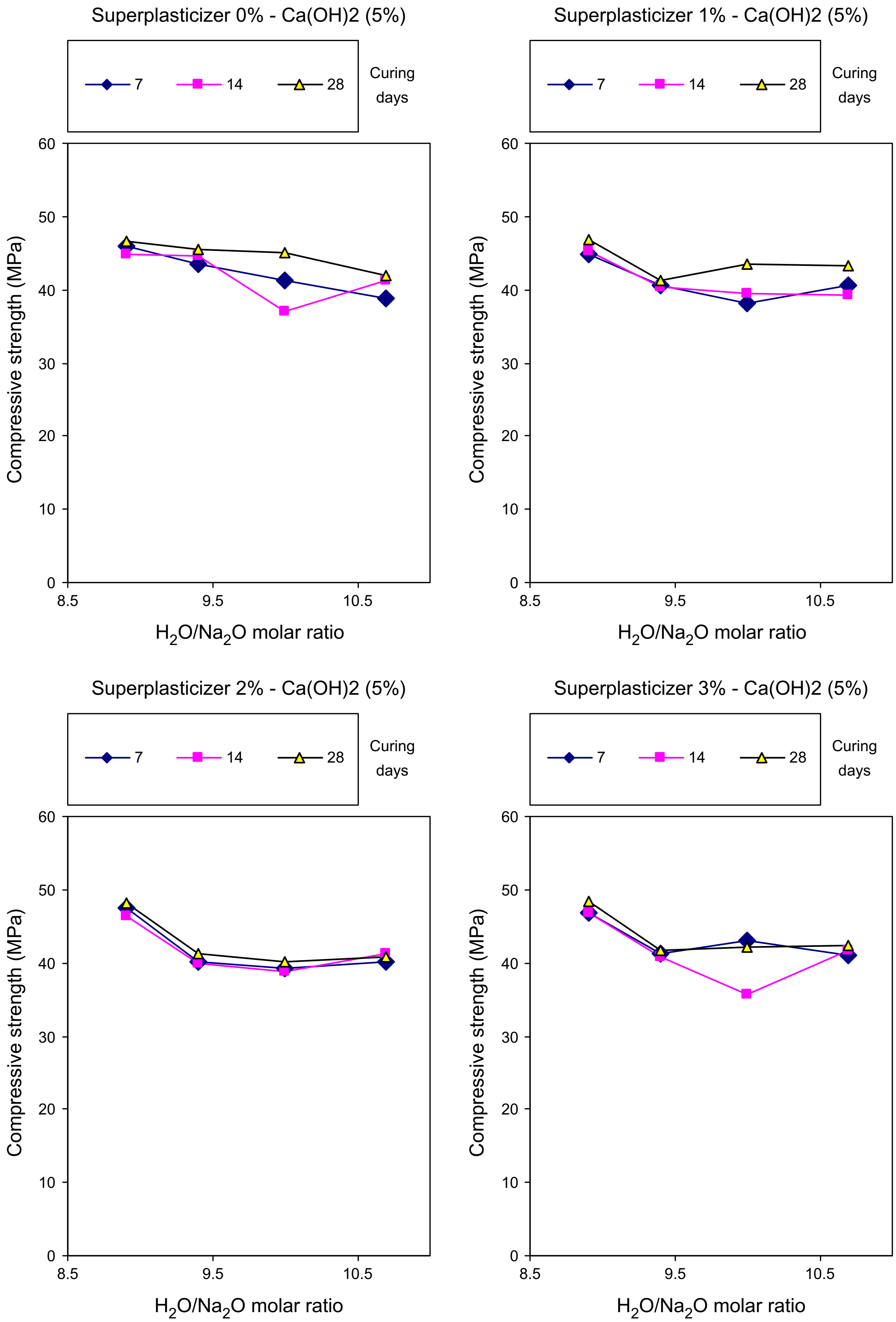

Fig. 11. Compressive strength versus H_2O/Na_2O atomic ratio for mortars with 5% of calcium hydroxide and several superplasticizer contents.

superplasticizer which needs 28 curing days to achieve a 40 MPa compressive strength, the other can easily achieved a high mechanical strength just after 7 days curing. Some of them can even reach 50 MPa. Figs. 10–12 show the compressive strength

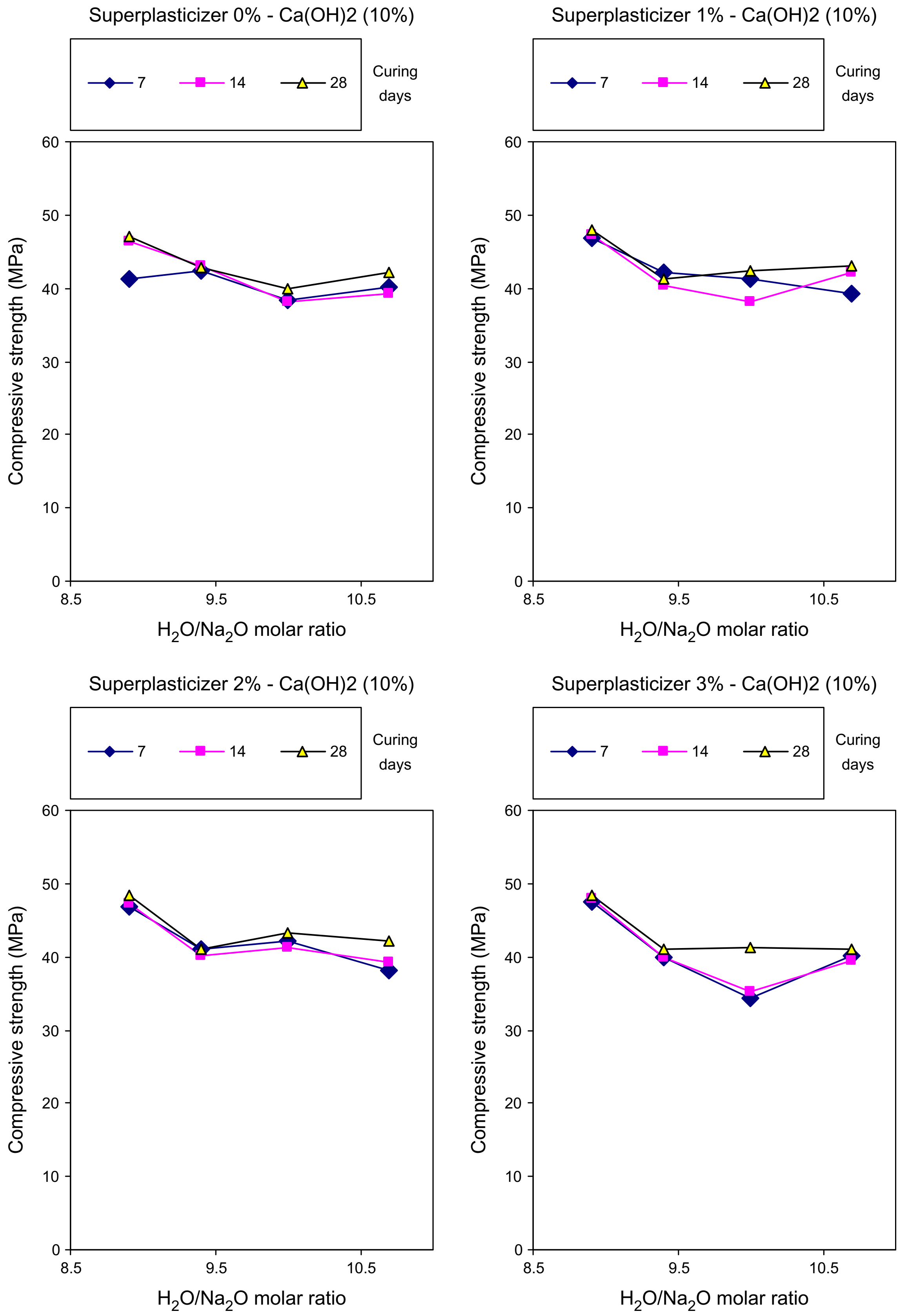

Fig. 12. Compressive strength versus H_2O/Na_2O atomic ratio for mortars with 10% of calcium hydroxide and several superplasticizer contents.

versus H_2O/Na_2O atomic ratio according to curing time. The results show a compressive strength increase with the H_2O/Na_2O molar ratio decrease below 9.5. This behaviour is independent of the calcium hydroxide content. Although other authors [15] obtained a

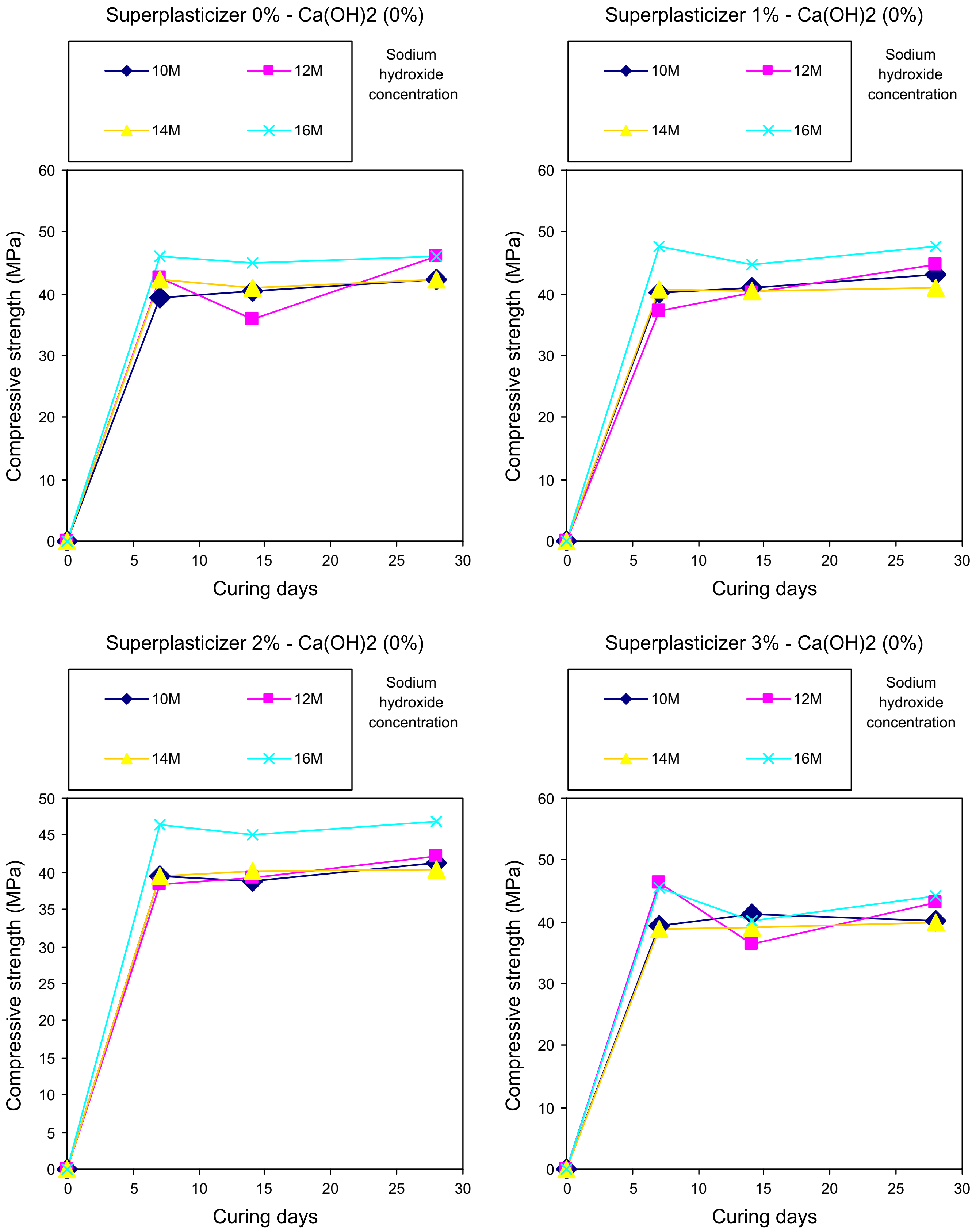

Fig. 13. Flexural strength according to age for mortar mixtures with 0% of calcium hydroxide and several sodium hydroxide concentrations and superplasticizer contents.

lower compressive strength for the same sodium hydroxide concentration and calcium hydroxide content (30 MPa for 16 M and 10% lime), when using tungsten mine waste mud the explanation for that is not entirely related to the different reactivity between

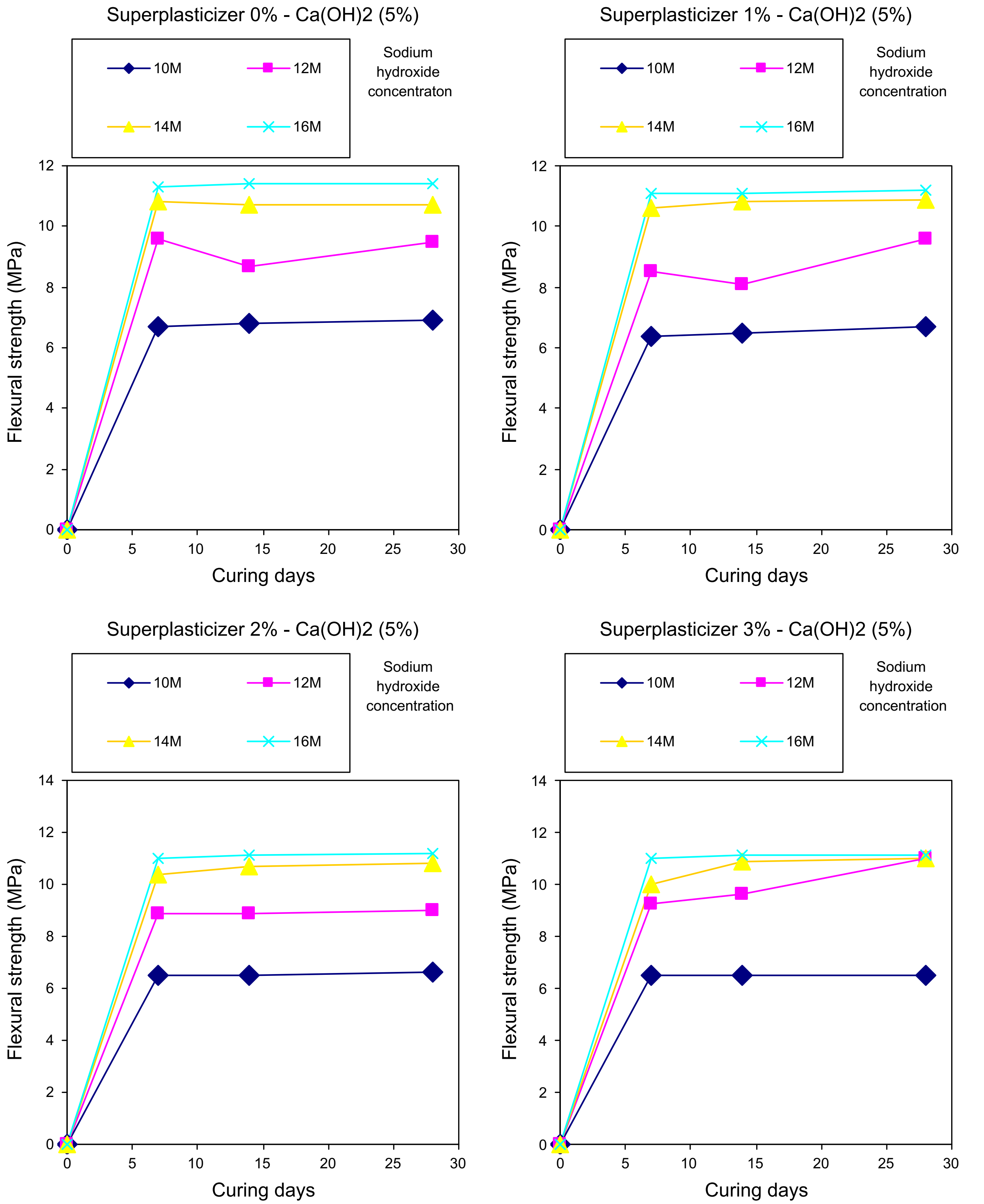

Fig. 14. Flexural strength according to age for mortar mixtures with 5% of calcium hydroxide and several sodium hydroxide concentrations and superplasticizer contents.

the metakaolin and the tungsten mine waste mud. The different results are much more related to the H_2O/Na_2O parameter which is 8.9 (16 M) in the present study and was 13.4 for those authors. When they used a H_2O/Na_2O around 10 they observed a compres-

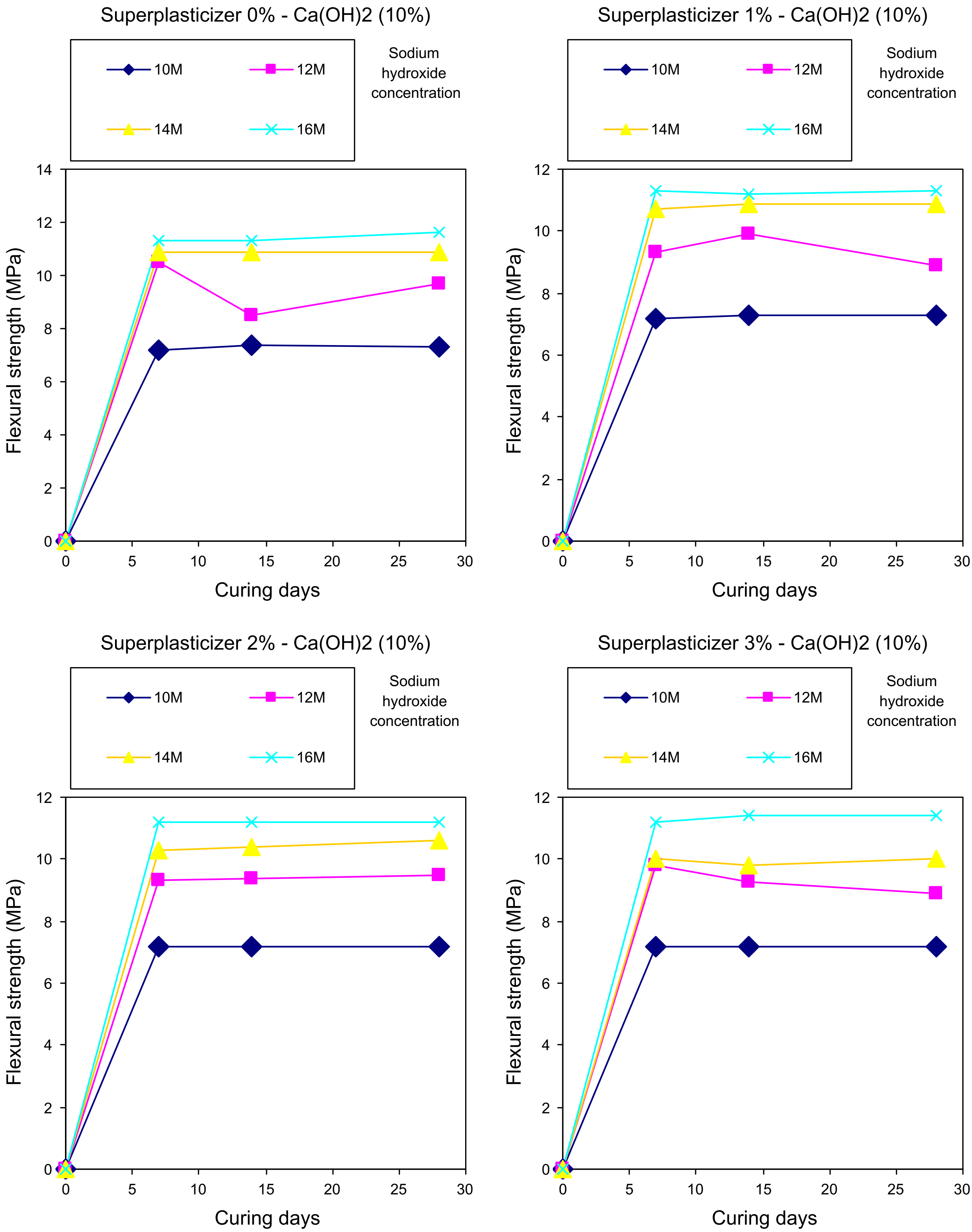

Fig. 15. Flexural strength according to age for mortar mixtures with 10% of calcium hydroxide and several sodium hydroxide concentrations and superplasticizer contents.

sive strength of 55 MPa after just 7 days curing. The flexural strength of alkali-activated mortars according to the curing days is showed in Figs. 13–15. The results are rather high between 8 and 12 MPa representing 20–25% of the compressive strength.

Similar findings had already been reported [24]. This is quite different from the behaviour observed for Portland cement based concrete for which flexural strength represents 10–15% of the compressive strength and a flexural strength above 10 MPa is uncommon. The results also show that flexural strength increased with and increased in sodium hydroxide concentration, the increase is similar to the increase already observed for the compressive strength in both cases that influence means an increase around 35%.

4. Conclusions

The results also show that the mortars workability is reduced with the increase of the sodium hydroxide concentration and also with a high replacement of metakaolin with calcium hydroxide, because metakaolin has a high Blaine fineness. The results also show that the compressive strength and flexural strength increased with and increased in sodium hydroxide concentration, in both cases that influence means an increase around 35%. The use of a superplasticizer content of 3% combined with a calcium hydroxide content of 10% is responsible for an increase in mortar flow from less than 50% to over 90% while maintaining a high level of mechanical strength. The results show that the use of a superplasticizer content up to 3% does not lead to mechanical strength reductions, with the exception of the mixture with a calcium hydroxide content of 10% and a sodium hydroxide concentration of 12 M.

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