Short-Term Compressive Strength of Fly Ash and Waste Glass Alkali-Activated Cement-Based Binder Mortars with Two Biopolymers

Z. Abdollahnejad[1]; M. Kheradmand[2]; and F. Pacheco-Torgal[3]

Abstract: The Roadmap to a Resource Efficient Europe aims that by 2020, waste will be managed as a resource. Thus materials that have the ability for the reuse of several types of wastes, such as alkali-activated cement-based binders (AACBs), will merit special attention. Some wastes like fly ash deserve special attention because they are generated in high amounts and have a very low reuse rate. This paper reports experimental results regarding the influence of the mix design of fly ash and waste glass AACB mortars containing two different biopolymers (carrageenan and xanthan) on their short-term mechanical performance. Microstructure and cost analysis are also included. The results show that a mixture of 80% fly ash, 10% waste glass, and 10% calcium hydroxide activated with an alkaline activator has the highest compressive strength. The results also show that the mortars with minor biopolymer carrageenan content are associated with a relevant increase in compressive strength and that the use of 0.1% of carrageenan leads to optimum compressive strength in most mixtures. The use of xanthan shows no beneficial effects on the compressive strength of AACB mortars. Several mixtures with xanthan even show a reduction in the compressive strength. **DOI: 10.1061/(ASCE)MT.1943-5533.0001920.** *© 2017 American Society of Civil Engineers.*

Author keywords: Waste reuse; Fly ash; Waste glass; Alkali-activated cement-based binders (AACB); Biopolymers; Mechanical strength; Cost analysis.

Introduction

The flagship initiative Roadmap to a Resource Efficient Europe has proven that resource efficiency is very important not only from an environmental point of view but also to boost industrial competitiveness in the EU. According to the Roadmap, "By 2020, waste is managed as a resource" [COM 571 (COM 2011)]. This is of paramount importance in the European context of a circular economy and zero waste target that may lead to an overall savings potential of 630 billion Euro per year for European industry and can also create more than 180,000 direct jobs in the EU by 2030 [COM 398 (COM 2014)]. Thus materials that have the ability for the reuse of several types of wastes, such as alkali-activated cement-based binders (AACBs) (Fig. 1), will merit special attention. AACBs are produced though the reaction of an aluminosilicate precursor with an activator, usually composed of hydroxide, silicate, carbonate, or sulfate, leading to the formation an amorphous aluminosilicate gel and secondary nanocrystalline zeolitelike structures. The activation of higher calcium content–based precursors, which are not classified as geopolymers, generates C-A-S-H gel that can coexist with minor secondary N-A-S-H gel (Provis 2014). These materials show important features but also some shortcomings that need to be addressed in order to compete against portland cement (Provis et al. 2008; Pacheco-Torgal et al. 2012, 2016). AACBs have a particular ability for the reuse of several types of wastes (Payá et al. 2014; Chinprasirt and Cao 2014; Bernal et al. 2016). Some wastes like fly ash deserve special attention because they are generated in high amounts and have a very low reuse rate. The United States has a reuse rate for fly ash of around 50%, meaning that 30 million tons of fly ash are not reused annually (ACAA 2016). Also recent studies (Bernal et al. 2016) concerning waste geopolymerization confirm that India and the Middle East currently report fly ash utilization rates below 20%. Other recent works confirm the importance of further studies regarding the development of geopolymers based on fly ash precursors (Zhuang et al. 2016). Waste glass is also a waste that is generated in relevant quantities and that merits increased recycling efforts. In Portugal approximately 425,000 t of waste glass were produced and only 192,000 t of them were recycled. Approximately 373 t of waste glass are generated daily in Hong Kong. According to some authors (Rashad 2014; Yu et al. 2016), the total amount of waste glass generated in the EU-27 in 2007 was 25.8 Mt. The use of waste glass in alkali-activated binders is especially interesting because its high SiO_2 content allows for a reduction on the content of sodium silicate, thus reducing the cost of this binder, which constitutes one of the AACBs' shortcomings (Pacheco-Torgal et al. 2016). This reason justified recent studies on alkali-activated materials containing waste glass (Redden and Neithalath 2014; Martinez-Lopez and Escalante-Garcia 2016; Wang et al. 2016). In this context, this paper reports results of compressive strength, X-ray diffraction (XRD), scanning electron microscope (SEM), and cost analyses of several fly ash and waste glass AACB mortars.

Experimental Program

Materials and Mix Design

The AACB mortars were prepared by mixing fly ash, calcium hydroxide (CH) milled waste glass (MG), fine aggregate, and

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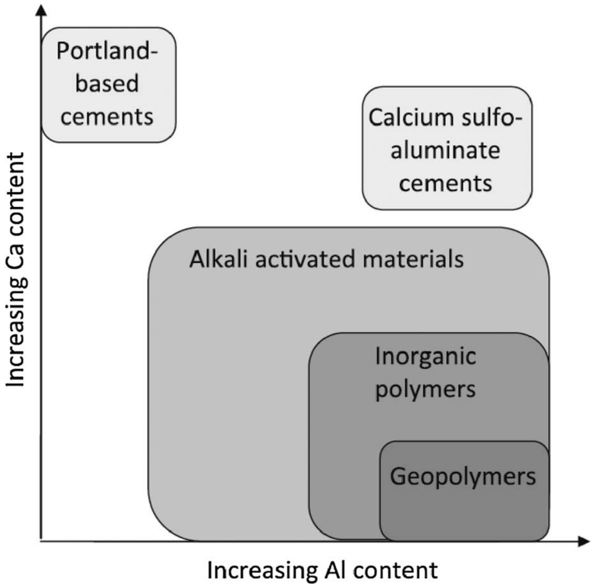


Fig. 1. Classification of different subsets of AAMs, with comparisons to OPC and calcium sulfoaluminate binder chemistry (reprinted from Springer, *Waste and Biomass Valorization*, "Chemical Research and Climate Change as Drivers in the Commercial Adoption of Alkali Activated Materials," Vol. 1, Issue 1, pp. 145–155, 2010, J. Van Deventer, J. Provis, P. Duxson, and D. Brice, with permission of Springer)

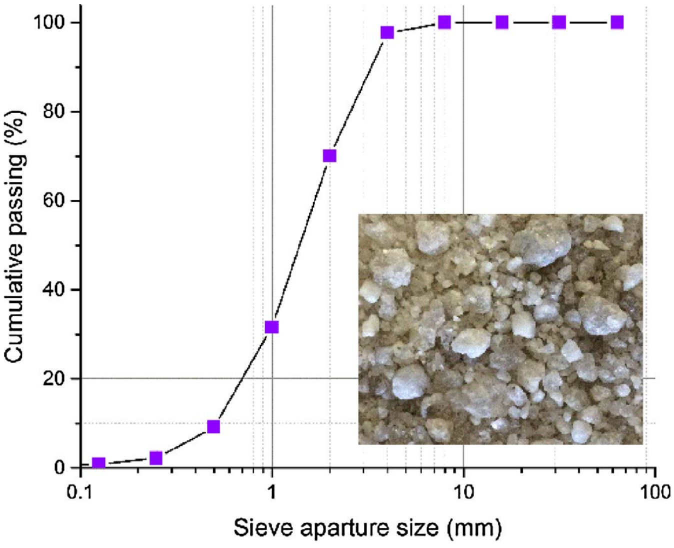


Fig. 2. Distribution of particle size of aggregates (image by M. Kheradmand)

Table 1. Chemical Composition and Physical Properties of Fly Ash

Parameter	% by weight
Composition	
SiO_2	60.81
Al_2O_3	22.68
Fe_2O_3	7.64
MgO	2.24
Na_2O	1.45
CaO	1.01
TiO_2	1.46
K_2O	2.70
Physical properties	
Specific gravity	2.30
Specific surface (cm^2/g)	3,430

an alkaline activator. The sand was used as inert filler provided from MIBAL (Minas de Barqueiros, S.A. Portugal). The sand was sieved (4.75- and 0.6-mm mesh) in order to remove dust particles. Fig. 2 shows the particle size of the sand. A sand:binder ratio of 4 was used. The fly ash was obtained from the PEGO Thermal Power Plant in Portugal and it was classified as Class F according to the ASTM-C618 standard (ASTM 2015). Table 1 presents the chemical composition and physical properties of fly ash. The sodium silicate liquid was supplied by MARCANDE (Portugal). The calcium hydroxide used in this study was a commercial Portuguese one (Lusical H100) with a chemical composition of $Ca(OH)_2 \geq 93\%$ and $MgO \leq 3$. Milled glass was used to partially replace fly ash. Waste glass powder was prepared from glass bottles, where the bottles were ground for 1 h in a ball mill in the laboratory. The density for the milled glass was $1.27\ g/cm^3$. The alkaline activator was prepared by mixing NaOH solution and sodium silicate solution in different mass ratios: 1, 1.5, and 2. An alkaline activator: binder ratio of 0.8 was used. The chemical composition of the sodium silicate was of 13.5% Na_2O, 58.7% SiO_2, and 45.2% H_2O. Solid sodium hydroxide were obtained from commercially available product of ERCROS (S.A., Spain). Distilled water was used to dissolve the sodium hydroxide flakes to avoid the effect of unknown contaminants in the mixing water. The sodium hydroxide (NaOH) solution was prepared by dissolving NaOH pellets in water to obtain concentrations of 4 and 8 mol/L cooled at room temperature. Two biopolymers, carrageenan and xanthan, were provided by Melbourne Food Depot (2016). Xanthan is a natural polysaccharide, which is produced by a biotechnological process involving fermentation of glucose or sucrose by the *Xanthomonas campestris* bacterium. Carrageenan is a natural high-molecular-weight polysaccharide, a purified extract from red seaweed. Different contents of biopolymers were used in the mix compositions, including 0.05, 0.1, and 0.15% (in weight of binder). The mix compositions are listed in Table 2.

Production, Testing, and Cost Analysis

During production, fly ash was mixed with fine aggregate, calcium hydroxide, milled waste glass, and biopolymer powder for 2 min. Then, the alkali activator solution was added and mixed for 5 min. Mortars were cast into cubic molds ($50 \times 50 \times 50\ mm^3$). After 24 h, specimens were demolded and cured in four different curing conditions, including (1) curing at ambient temperature, (2) thermal curing at a temperature of 60°C for 1 h, (3) thermal curing at a temperature of 60°C for 2 h, and (4) thermal curing at a temperature of 60°C for 3 h. After exposing the specimens to heat treatment, specimens were cured in laboratory conditions (25°C and 65% relative humidity). Compressive strength testing was carried out at 7 days with respect to the recommendations of the European standard EN1015-11 (CEN 1999). XRD analysis was employed to assess the crystalline structure of the mixtures. Powder XRD was implemented on a Bruker D8 Discover with Cu-Kα radiation ($\lambda = 154{,}060$ Å) at 40 kV and 40 mA. Each sample was scanned from 5 to 70° at a speed of 0.4°/s. The analysis for phase identification was performed using analytical software. Cost analysis of the mortars was carried out. The microstructural observation for several AACB mixtures was carried out using standard SEM microscopy (NOVA 200 Nano SEM). Micrographs and chemical compositions were collected at accelerating voltages of 10 and 15 kV, respectively, and variable working distance from 6 to

Table 2. Mix Proportions of Geopolymer Mortars (kg/m^3)

Mix composition	Fly ash	CH	MG	Xanthan	Carrageenan	Sand	SS	SH	Molarity (mol/L)
90FA_10CH_4M_2SS/SH_0.8A/B	379	42.0	—	—	—	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B	379	42.0	—			1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B	332	41.6	41.6			1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B	308	41.1	61.7			1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B	285	40.7	81.5			1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B	379	42.0	—			1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B	379	42.0	—			1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B	332	41.6	41.6			1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B	309	41.1	61.7			1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B	285	40.7	81.5			1,630	163	163	
90FA_10CH_4M_2SS/SH_0.8A/B_0.05CAR	379	42.0	—		0.210	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B_0.05CAR	379	42.0	—		0.210	1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B_0.05CAR	332	41.6	41.6		0.208	1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.05CAR	308	41.1	61.7		0.205	1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.05CAR	285	40.7	81.5		0.200	1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B_0.05CAR	379	42.0	—		0.210	1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B_0.05CAR	379	42.0	—		0.210	1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.05CAR	332	41.6	41.6		0.208	1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.05CAR	309	41.1	61.7		0.205	1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.05CAR	285	40.7	81.5		0.200	1,630	163	163	
90FA_10CH_4M_2SS/SH_0.8A/B_0.1CAR	379	42.0	—	—	0.420	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B_0.1CAR	379	42.0	—		0.420	1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B_0.1CAR	332	41.6	41.6		0.410	1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.1CAR	308	41.1	61.7		0.410	1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.1CAR	285	40.7	81.5		0.400	1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B_0.1CAR	379	42.0	—		0.420	1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B_0.1CAR	379	42.0	—		0.420	1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.1CAR	332	41.6	41.6		0.410	1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.1CAR	309	41.1	61.7		0.410	1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.1CAR	285	40.7	81.5		0.400	1,630	163	163	
90FA_10CH_4M_2SS/SH_0.8A/B_0.15CAR	379	42.0	—	—	0.630	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B_0.15CAR	379	42.0	—		0.630	1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B_0.15CAR	332	41.6	41.6		0.624	1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.15CAR	308	41.1	61.7		0.617	1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.15CAR	285	40.7	81.5		0.611	1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B_0.15CAR	379	42.0	—		0.630	1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B_0.15CAR	379	42.0	—		0.630	1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.15CAR	332	41.6	41.6		0.624	1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.15CAR	309	41.1	61.7		0.617	1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.15CAR	285	40.7	81.5		0.611	1,630	163	163	
90FA_10CH_4M_2SS/SH_0.8A/B_0.05XAN	379	42.0	—	0.210	—	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B_0.05 XAN	379	42.0	—	0.210		1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B_0.05 XAN	332	41.6	41.6	0.208		1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.05 XAN	308	41.1	61.7	0.205		1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.05 XAN	285	40.7	81.5	0.200		1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B_0.05 XAN	379	42.0	—	0.210		1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B_0.05 XAN	379	42.0	—	0.210		1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.05 XAN	332	41.6	41.6	0.208		1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.05 XAN	309	41.1	61.7	0.205		1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.05 XAN	285	40.7	81.5	0.200		1,630	163	163	
90FA_10CH_4M_2SS/SH_0.8A/B_0.1 XAN	379	42.0	—	0.420	—	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B_0.1 XAN	379	42.0	—	0.420		1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B_0.1 XAN	332	41.6	41.6	0.410		1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.1 XAN	308	41.1	61.7	0.410		1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.1 XAN	285	40.7	81.5	0.400		1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B_0.1 XAN	379	42.0	—	0.420		1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B_0.1 XAN	379	42.0	—	0.420		1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.1 XAN	332	41.6	41.6	0.410		1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.1 XAN	309	41.1	61.7	0.410		1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.1 XAN	285	40.7	81.5	0.400		1,630	163	163	

Table 2. (*Continued*.)

Mix composition	Fly ash	CH	MG	Xanthan	Carrageenan	Sand	SS	SH	Molarity (mol/L)
90FA_10CH_4M_2SS/SH_0.8A/B_0.15 XAN	379	42.0	—	0.630	—	1,685	269	134	4
90FA_10CH_4M_1SS/SH_0.8A/B_0.15 XAN	379	42.0	—	0.630		1,685	169	169	
80FA_10CH_10MG_4M_2SS/SH_0.8A/B_0.15 XAN	332	41.6	41.6	0.624		1,664	222	111	
75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.15 XAN	308	41.1	61.7	0.617		1,647	198	132	
70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.15 XAN	285	40.7	81.5	0.611		1,630	163	163	
90FA_10CH_8M_2SS/SH_0.8A/B_0.15 XAN	379	42.0	—	0.630		1,685	269	134	8
90FA_10CH_8M_1SS/SH_0.8A/B_0.15 XAN	379	42.0	—	0.630		1,685	169	169	
80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.15 XAN	332	41.6	41.6	0.624		1,664	222	111	
75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.15 XAN	309	41.1	61.7	0.617		1,647	198	132	
70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.15 XAN	285	40.7	81.5	0.611		1,630	163	163	

Note: SH = sodium hydroxide; SS = sodium silicate.

Table 3. Costs of the Materials Used in the Geopolymer Mix Compositions and the Energy Used for Heating

Parameter (Euro/kg)	Value
Aggregate	0.02
MG	0.009
CH	0.30
Fly ash	0.03
Sodium silicate	0.29
Sodium hydroxide	0.85
Thermal curing	0.05
Carrageenan	105.00
Xanthan	84.00

8 mm. The cylindrical subsamples from Zones 1 to 3 were coated with a 30-nm layer of gold-palladium (60% gold and 40% palladium), and then the SEM/EDS examination for all the specimens was conducted. The costs of the materials that were used to calculate the cost of the mortars are listed in Table 3.

Results and Discussion

Compressive Strength at Ambient Temperature

The results of the compressive strength after 7 days curing of AACB mixtures at ambient temperature without biopolymers are shown in Fig. 3. The mixture 80FA_10CH_10MG_2SS/SH_0.8A/B shows the highest compressive strength of around 9 MPa. This mechanical performance is high enough for several construction applications, such as use in renders and for masonry units, for both the 4M and 8M sodium hydroxide concentrations. The explanation lies in the fact that for low sodium hydroxide concentrations the main hydration product formed is a C-S-H gel (Alonso and Palomo 2001, 2001a; Garcia-Lodeiro et al. 2013, 2016). The replacement of 10% fly ash by milled waste glass shows a relevant increase in the short-term compressive strength of around 30%. The waste glass provides reactive silica that contribute to the increase of the compressive strength. The reduction of the sodium silicate:sodium hydroxide ratio from 2 to 1 shows no influence on the compressive strength for both sodium hydroxide concentrations. Increasing the replacement of fly ash with milled glass from 10 to 20% by weight, as well as decreasing the ratio of sodium silicate to sodium hydroxide from 2 to 1, reduced the compressive strength of the mix composition of 80FA_10CH_10MG_4M_2SS/SH_0.8A/B by about 30%. This is because silica from waste glass is not as reactive as liquid Na_2SiO_3.

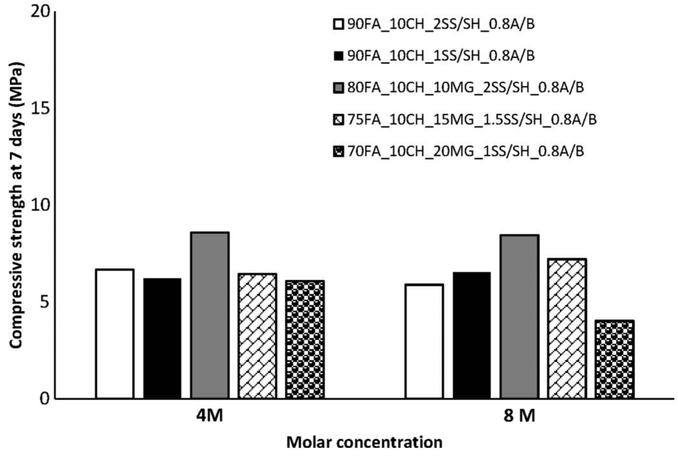


Fig. 3. Compressive strength of AACB mortars without biopolymers after 7 days of curing

Compressive Strength of Mixtures with Biopolymers Cured at Ambient Temperature

Fig. 4 shows the compressive strength of AACB mixtures with the biopolymer carrageenan cured at ambient temperature over 7 days. The biopolymer carrageenan increased compressive strengths of mixtures 90FA_10CH_2SS/SH_0.8A/B and 90FA_10CH_1SS/SH_0.8A/B. This increment was higher for these mixtures with 4M sodium hydroxide concentration. Because of the addition of carrageenan, the maximum compressive strength and the maximum increase of compressive strength were about 17 MPa and 3 times, respectively, for the mixture 90FA_10CH_2SS/SH_0.8A/B with 0.15% of carrageenan. For mixtures 80FA_10CH_10MG_2SS/SH_0.8A/B and 75FA_10CH_15MG_2SS/SH_0.8A/B with molar concentrations of 4 mol/L, the compressive strength increased by increasing the content of carrageenan up to 0.1%, while increasing the content of carrageenan to 0.15% reduced the compressive strength. Conversely, by increasing the molar concentration from 4 to 8 mol/L, increasing the content of carrageenan up to 0.15% resulted in recording the maximum compressive strength (16.2 MPa for both mixtures 80FA_10CH_10MG_2SS/SH_0.8A/B and 75FA_10CH_15MG_1.5SS/SH_0.8A/B). As illustrated in Fig. 4(e), by increasing the replacement of fly ash with milled glass up to 20% by weight, as well as reducing the sodium silicate:sodium hydroxide ratio to 1 in the mix composition of 70FA_10CH_20MG_1SS/SH_0.8A/B, the rate of the compressive strength increment was slightly reduced. Moreover, increasing the molar concentration from 4 to 8 mol/L has no significant effect on increasing the compressive

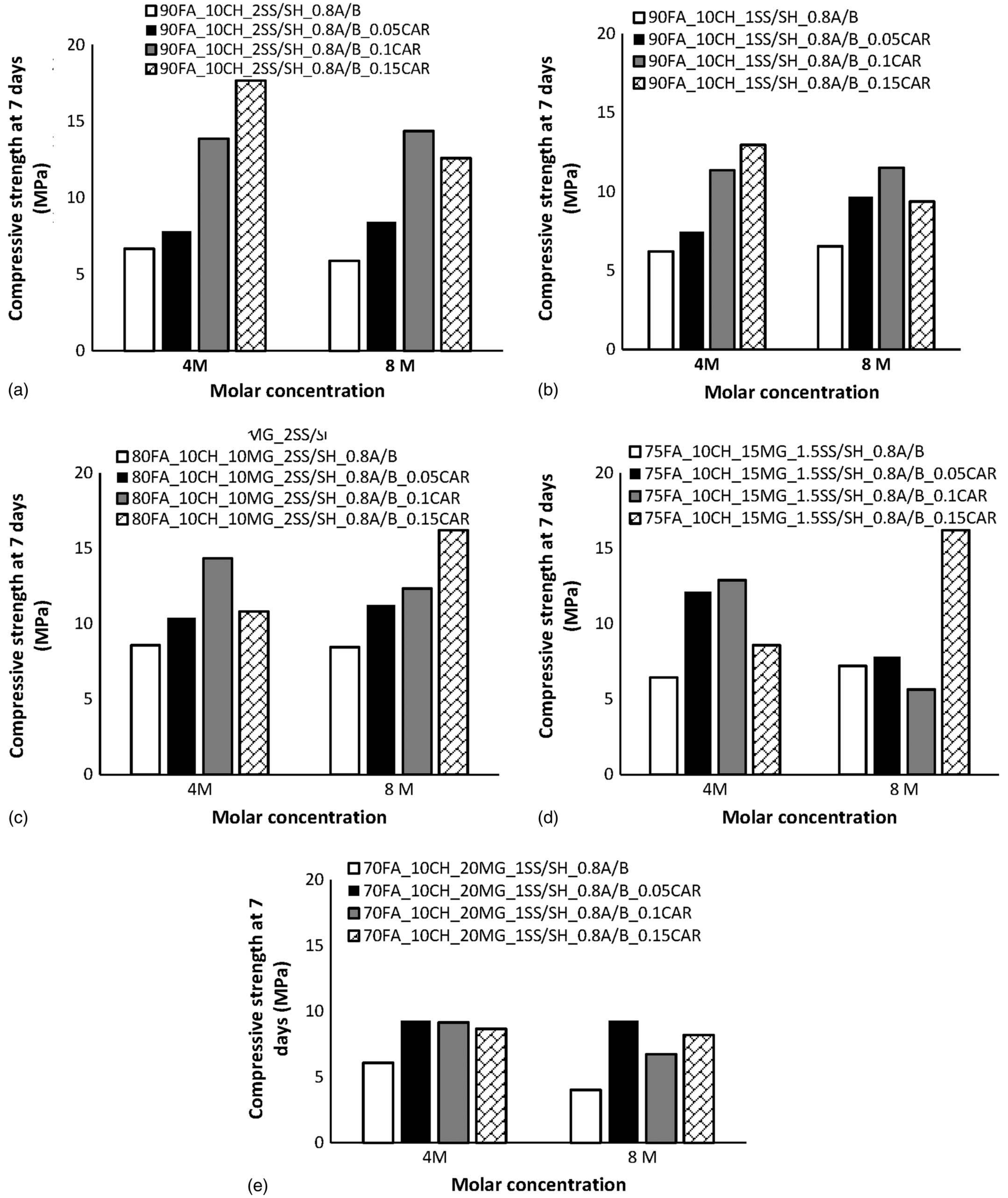


Fig. 4. Compressive strength of AACB mixtures with biopolymer carrageenan: (a) 90FA_10CH_2SS/SH_0.8 A/B; (b) 90FA_10CH_1SS/SH_0.8 A/B; (c) 80FA_10CH_10MG_2SS/SH_0.8 A/B; (d) 75FA_10CH_10MG_1.5SS/SH_0.8 A/B; (e) 70FA_10CH_20MG_1SS/SH_0.8 A/B

strength of the mix composition of 70FA_10CH_20MG_1SS/SH_0.8A/B. Some authors who studied geopolymers with carrageenan have reported an increase in compressive strength with biopolymer content with a peak at 0.1% (Li and Zhang 2016). This confirms that the use of 0.1% of carrageenan leads to optimum compressive strength. These authors suggested that the carrageenan coated and bridged the fly ash particles and led to the formation of a more condensed structure, improving the mechanical behavior of

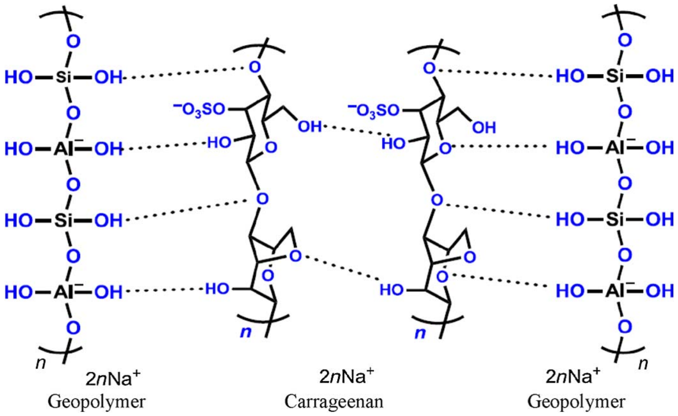


Fig. 5. Hydrogen bonds (represented by dashed lines) formed between carrageenan and fly ash–based geopolymer as well as within kappa-carrageenan macromolecules (reprinted from *Biopolymers and Biotech Admixtures for Eco-Efficient Construction Materials*, Z. Li and L. Zhang, "Fly ash-based geopolymer with kappa-carrageenan biopolymer," pp. 173–192, Copyright 2016, with permission from Elsevier)

the biopolymer/geopolymer composite. For those authors, the primary effect of the incorporation of carrageenan into a geopolymer could be attributed to the presence of large amounts of hydrogen bonds in the biopolymer macromolecules (Fig. 5). Fig. 6 shows the compressive strength of AACB mixtures with biopolymer xanthan cured at ambient temperature over 7 days. Xanthan is not as effective as carrageenan at enhancing compressive strength. With the exception of 90FA_10CH_8M_1SS/SH_0.8A/B_0.1XAN, the addition of xanthan had no significant effect on increasing the compressive strength of mix compositions. The maximum compressive strength and increase of compressive strength as a result of the addition of xanthan were recorded to be about 13 MPa and 2 times, respectively, for mixture 90FA_10CH_8M_1SS/SH_0.8A/B_0.1XAN. Concerning the mixtures 80FA_10CH_10MG_2SS/SH_0.8A/B, 75FA_10CH_15MG_1.5SS/SH_0.8A/B, and 70FA_10CH_20MG_1SS/SH_0.8A/B, the addition of xanthan biopolymer actually reduced their compressive strengths.

Compressive Strength of Mixtures with Biopolymers and Precuring

Fig. 7 shows the compressive strength of AACB mortars activated with 4M and with carrageenan using a precuring temperature of 60°C over several hours, while Fig. 8 shows the results for the same mixtures activated with an 8M sodium hydroxide concentration. Figs. 9 and 10 show the compressive strength of AACB mortars with xanthan. There is a general trend of strength increase with curing time. Several authors showed that precuring of alkali-activated binders before they are subjected to normal curing has been proven to further enhance their compressive strength (Kim and Kim 2013; Atis et al. 2015; Ming et al. 2016). Temperature helps to increase the dissolution of silica and aluminum from aluminosilicate precursors and accelerates the alkaline activation and the hardening process. However, some have reported a compressive strength loss (Redden and Neithalath 2014). On the other hand, this requires energy that is costly and has obvious environmental effects. Several temperatures and several exposure times were found for different mixtures and recently Gorhan et al. (2016) reported an ideal curing temperature and curing time of 60°C and 2 h, respectively, for the production of the alkali-activated paste based on fly ash and metakaolin. For AACB mortars activated with 4M and with carrageenan biopolymer, the maximum compressive strength was 10.78 MPa for the mixture 80FA_10CH_10MG_4M_2SS/SH_0.8A/B exposed to 60°C for 3 h, while the maximum increase of the compressive strength was detected to be about 43% for the mixture 75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B with exposure to 3 h of thermal curing. A reduction in the compressive strength of mixtures 90FA_10CH_4M_2SS/SH_0.8A/B, 90FA_10CH_4M_1SS/SH_0.8A/B, and 80FA_10CH_10MG_4M_2SS/SH_0.8A/B was reported. A maximum compressive strength of 16.18 MPa was obtained for mixture 75FA_10CH_15MG_4M_1.5SS/SH_0.8 A/B with the addition of 0.05% carrageenan and curing at 60°C for 3 h, as depicted in Fig. 7(b). The maximum increase of compressive strength measured about 33% for 3 h of thermal curing in the mix composition of 75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B, while the maximum reduction was detected to be about 14% with 1 h of thermal curing for the mix composition of 80FA_10CH_4M_2SS/SH_0.8A/B. Regarding the results indicated in Fig. 7(c), using the thermal curing had no significant effects on increasing the compressive strength of mixtures containing 0.1% carrageenan. The maximum increase and decrease of compressive strength as a result of thermal curing measured about 6 and 9% for mix compositions of 70FA_10CH_20MG_4M_1SS/SH_0.8A/B with 1 h of heating and 90FA_10CH_4M_1SS/SH_0.8A/B with 2 h of heating, respectively. Replacing 10% fly ash with milled glass (80FA_10CH_10MG_4M_2SS/SH_0.8A/B) slightly increased the compressive strength of the specimens heated up to 2 h at 60°C, so that the maximum compressive strength was recorded to be 14.59 MPa under 1 h of heating. With respect to the results illustrated in Fig. 7(d), thermal curing has no specific effect on increasing or decreasing the compressive strengths of the mix compositions containing 0.15% carrageenan. Use of thermal curing for the mixtures 90FA_10CH_4M_2SS/SH_0.8A/B_0.15CAR and 90FA_10CH_4M_1SS/SH_0.8A/B_0.15CAR reduced the compressive strength in comparison to ambient temperature curing. The reduction of compressive strength in the mixture 90FA_10CH_4M_0.8A/B_0.15CAR was intensified by decreasing the ratio of sodium silicate to sodium hydroxide from 2 to 1. Additionally, regardless of the curing time at 60°C, it was obtained that replacing 10% fly ash with milled glass in the mix composition of 80FA_10CH_10MG_4M_2SS/SH_0.8A/B reduced the compressive strength of the specimens, when compared to the mix composition of 90FA_10CH_4M_2SS/SH_0.8A/B. By increasing the replacement of fly ash with milled glass and also reducing the ratio of sodium silicate to sodium hydroxide, the compressive strength was reduced, while thermal curing increased the compressive strengths of these mix compositions compared to the compressive strengths of specimens cured at ambient temperature. The maximum increase and decrease of compressive strength were recorded to be about 41 and 11% in the mix compositions of 75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.15CAR with 3 h of heating and 90FA_10CH_4M_2SS/SH_0.8A/B_0.15CAR with 1 h of heating, respectively. The compressive strength of the mixture 90FA_10CH_8M_2SS/SH_0.8A/B increased regardless of curing time. However, reducing the sodium silicate:sodium hydroxide ratio from 2 to 1 and exposing specimens to under 1 h of heating decreased the compressive strength of the mix composition of 90FA_10CH_1SS/SH_0.8A/B. Increasing the time of thermal curing from 1 h to 2 and 3 h for the mix composition of 90FA_10CH_8M_1SS/SH_0.8A/B increased the polymerization process and subsequently increased the compressive strength. Interestingly, it was found that replacing 10% fly ash with milled glass in the mix composition of 80FA_10CH_10MG_8M_2SS/SH_0.8A/B not only reduced the compressive strength for a precuring during 2 h and increased it for a longer procuring period. When increasing the replacement of fly ash with milled glass from 10 to 20% by weight and decreasing the ratio of

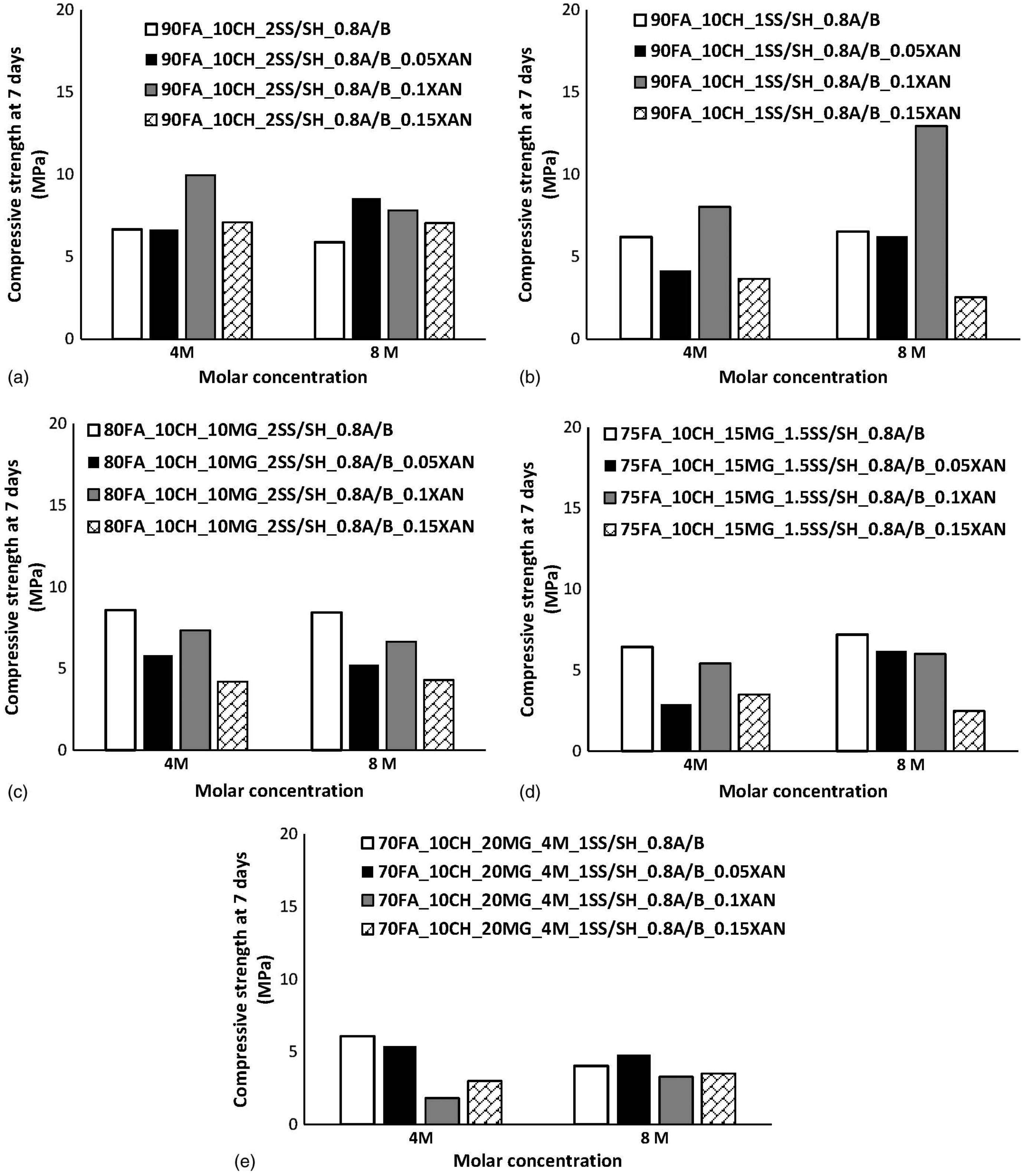


Fig. 6. Compressive strength of AACB mixtures with biopolymer xanthan: (a) 90FA_10CH_2SS/SH_0.8 A/B; (b) 90FA_10CH_1SS/SH_0.8 A/B; (c) 80FA_10CH_10MG_2SS/SH_0.8 A/B; (d) 75FA_10CH_10MG_1.5SS/SH_0.8 A/B; (e) 70FA_10CH_20MG_1SS/SH_0.8 A/B

sodium silicate to sodium hydroxide from 2 to 1, compressive strength decreased. Because of thermal curing, the maximum increase in the compressive strength was recorded to be about 45% in the mix composition of 90FA_10CH_8M_2SS/SH_0.8A/B with 3 h of heating. The maximum increase of the compressive strength was detected for mixture 90FA_10CH_8M_2SS/SH_0.8A/B cured over 3 h. Except for the mixture 75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.05CAR, curing all mix compositions with 3 h of heating increased the compressive strength of specimens in comparison to curing at ambient temperature. Curing the mix composition of 75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.05CAR for 1 h increased the compressive strength, while heating longer

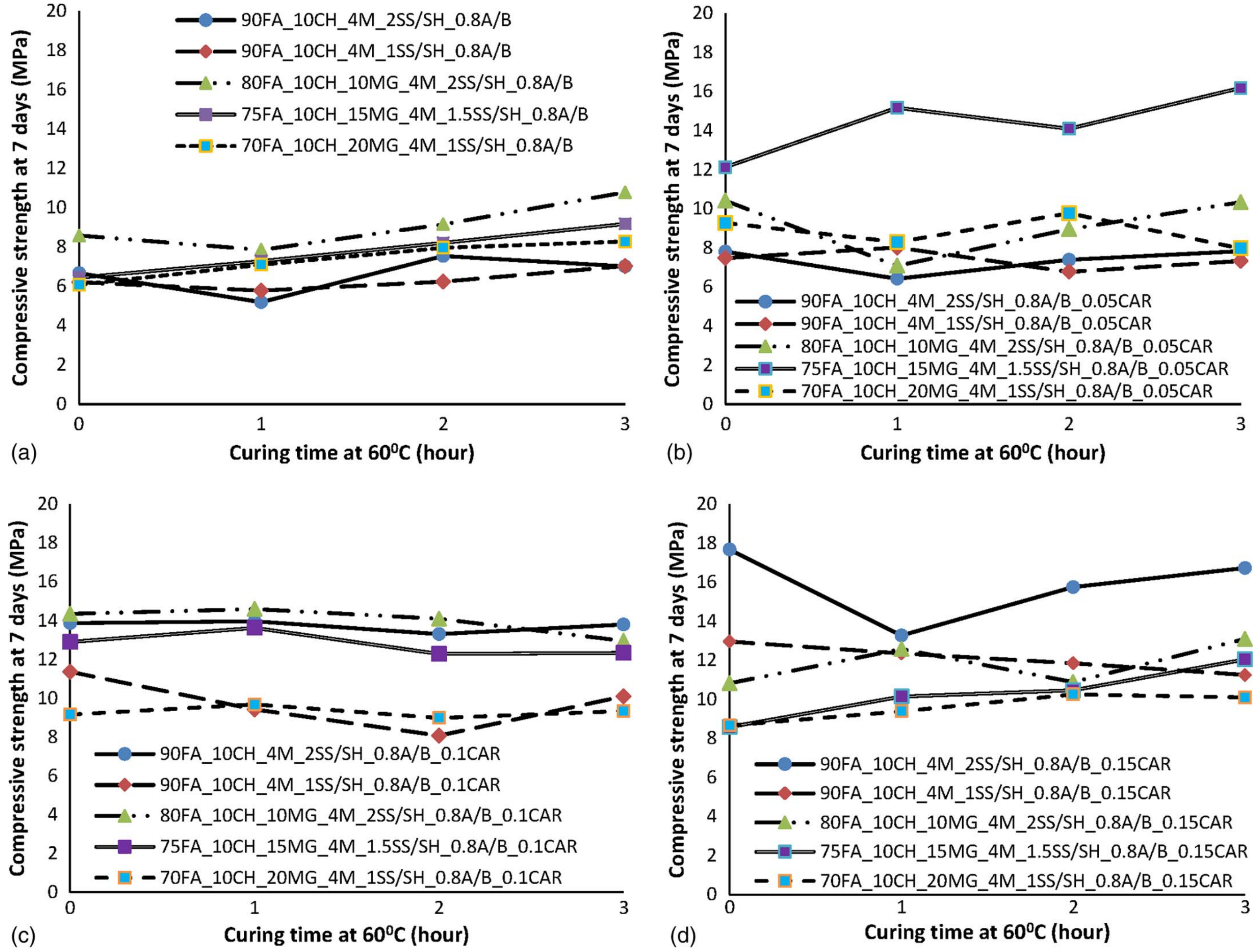


Fig. 7. Compressive strength of AACB mixtures cured at 60°C with 4M and the following biopolymer carrageenan content: (a) 0%; (b) 0.05%; (c) 0.1%; (d) 0.15%

than 1 h reduced the compressive strength. Figs. 8(c and d) show that an increased trend in the compressive strength of all compositions was obtained by increasing the content of carrageenan from 0.05 to 0.1 and 0.15% and increasing the curing time. Thus, the maximum compressive strength for mix compositions containing 0.1% carrageenan was obtained as 16 MPa in the mix composition of 90FA_10CH_8M_2SS/SH_0.8A/B. Furthermore, the maximum increase of the compressive strength was recorded to be about 50% for the mix composition of 75FA_10CH_15MG_8M_1.5SS/SH_0.8A/B_0.1CAR cured with 3 h of heating. As indicated in Fig. 8(d), by increasing the content of carrageenan from 0.1 to 0.15%, the maximum compressive strength was obtained to be about 19.50 MPa in the mix composition of 90FA_10CH_8M_2SS/SH_0.8A/B. Moreover, the maximum increase of the compressive strength was recorded to be about 62% for the mix composition of 90FA_10CH_8M_1SS/SH_0.8A/B_.015CAR, cured for 3 h. Based on the results obtained in Fig. 9(a), heating all mix compositions at 60°C over 3 h increased the compressive strength, when compared to the mix compositions cured at ambient temperature. The maximum compressive strength was recorded to be about 9 MPa for the mixture 70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.05XAN with 2 h of heating. Additionally, the maximum increase of compressive strength was recorded to be more than two times in the mix composition of 75FA_10CH_15MG_4M_1.5SS/SH_0.8A/B_0.05XAN as a result of 3 h of heating. When the content of xanthan is increased from 0.05 to 0.1% and 0.15%, thermal curing had no significant effects on increasing the compressive strengths of mix compositions, as indicated in Figs. 9(b and c). The maximum increases of the compressive strength of mix compositions containing 0.1 and 0.15% xanthan were registered to be about 10 and 46% for the mix composition of 70FA_10CH_20MG_4M_1SS/SH_0.8A/B_0.1XAN cured with 1 and 3 h of heating, respectively. By comparing the results indicated in Figs. 9 and 10, it was found that thermal curing results in the lower scattering compressive strengths in the mix compositions with molar concentrations of 4 than 8 mol/M. 3 h of heating increased the compressive strength of all mix compositions except for the mix composition of 70FA_10CH_20MG_8M_1SS/SH_0.8A/B_0.05XAN, as indicated in Fig. 10(a). Thermal curing not only increased the compressive strength for this mix composition, but also reduced it. The maximum compressive strength and the maximum increase of compressive strength were detected to be about 12.82 MPa and 60% for mix compositions of 90FA_10CH_2SS/SH_0.8A/B_0.05XAN cured with 2 h of heating and 80FA_10CH_10MG_2SS/SH_0.8A/B_0.05XAN

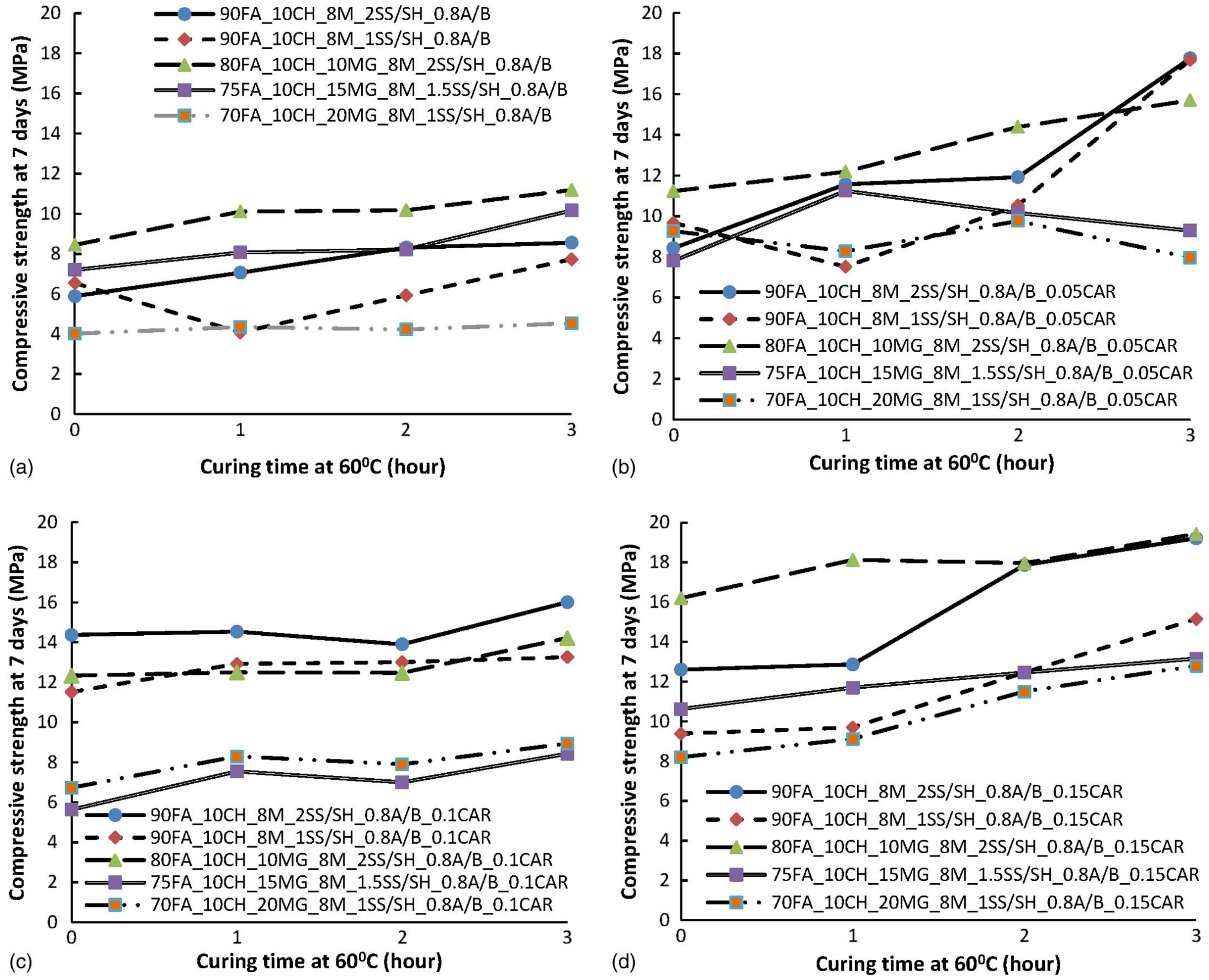


Fig. 8. Compressive strength of AACB mixtures cured at 60°C with 8M and to the following biopolymer carrageenan content: (a) 0%; (b) 0.05%; (c) 0.1%; (d) 0.15%

cured with 3 h of heating, respectively. Promisingly, replacing fly ash with milled glass from 10 to 15% by weight and reducing the sodium silicate:sodium hydroxide ratio from 2 to 1 did not decrease the compressive strength. By increasing the content of xanthan from 0.05 to 0.1%, in Fig. 10(b), heating increased the compressive strengths of all mix compositions, except for 90FA_10CH_8M_1SS/SH_0.8A/B_0.1XAN. The maximum compressive strength and the maximum increase of compressive strength were recorded to be about 13 MPa and 45% for mix compositions of 90FA_10CH_1SS/SH_0.8A/B_0.1XAN cured at ambient temperature and 80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.1XAN cured with 3 h of heating. Furthermore, it was interestingly observed that heating the specimens made with 80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.1XAN for more than a couple of hours caused the chemical reactions to become more rapid and subsequently enhanced the compressive strength, when compared to 90FA_10CH_8M_1SS/SH_0.8A/B_0.1XAN. The results in Fig. 10(c) indicate that thermal curing has no significant effects on increasing the compressive strength of all mix compositions containing 0.15% xanthan. The maximum increase of the compressive strength was obtained to be about 45% for the mix composition of 80FA_10CH_10MG_8M_2SS/SH_0.8A/B_0.15XAN cured with 2 h of heating.

SEM and XRD

Fig. 11 shows the microstructure of two AACB mortars with the biopolymer carrageenan at room temperature and after 3 h at 60°C. Figs. 11(a and b) are related to an AACB mortar mixture that showed no strength increase after thermal treatment. The explanation for that may be related to the microcracks shown in Fig. 11(b). The second mixture showed around a 45% strength increase after being exposed to 60°C over 3 h. The microstructure seems to confirm that because it appears to be denser. Fig. 12 shows XRD spectra of several AACB mortars. The C-(A)-S-H phase displays some structural similarity with aluminum-containing tobermorite (PDF#19-0052). Portlandite $[Ca(OH)_2]$ (PDF #44–1481) and ettringite $[Ca_6Al_2(SO4)_3(OH)_{12} \cdot 26H_2O]$ (PDF#41–1451) are also noticed. Small amounts of stilbite-Ca $(NaCa_2Al_5Si_{13}O_{36} \cdot 14H_2O)$

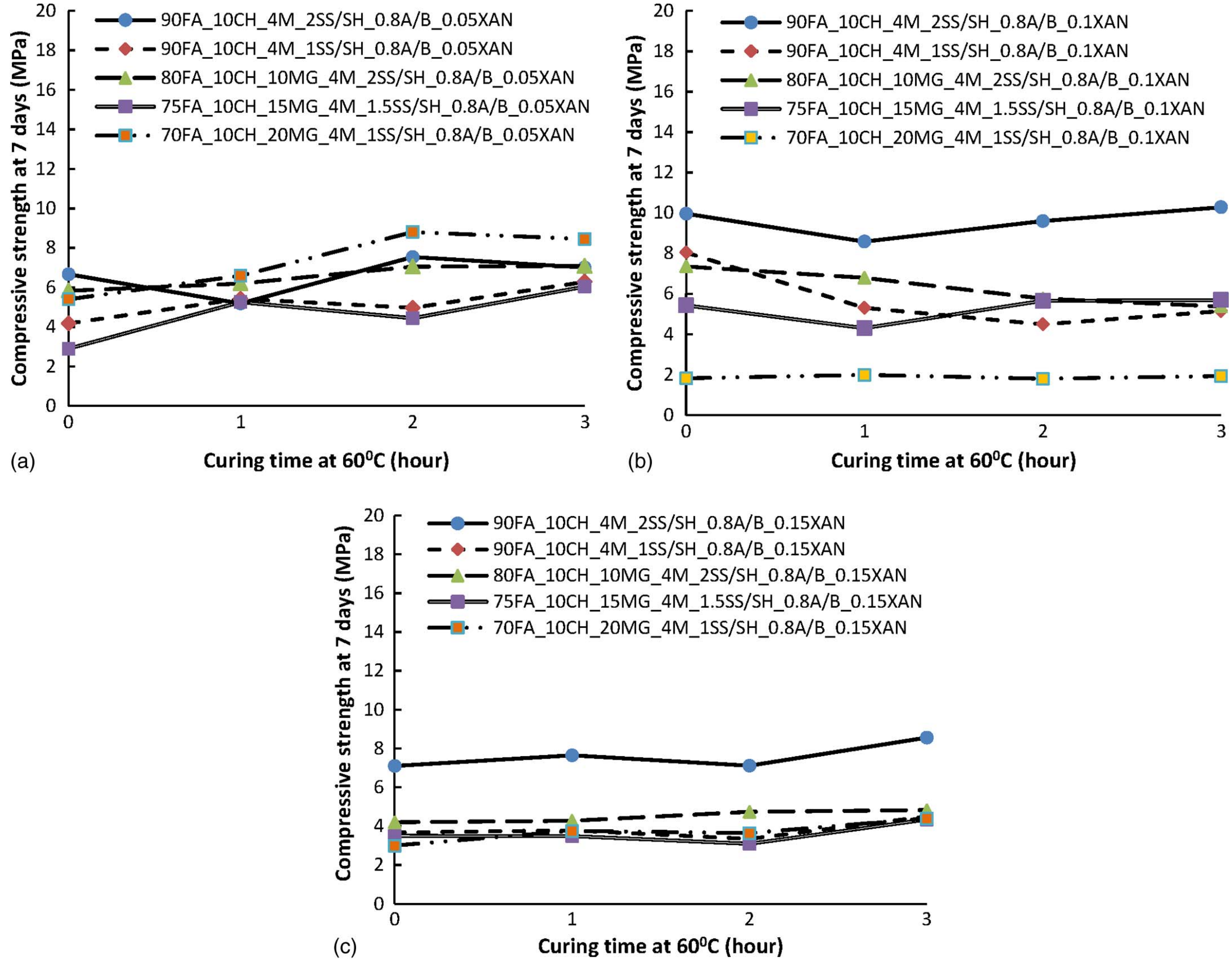


Fig. 9. Compressive strength of AACB mixtures cured at 60°C with 4M and the following biopolymer xanthan content: (a) 0%; (b) 0.05%; (c) 0.1%; (d) 0.15%

(PDF#44-1479) were detected. Vaterite (PDF#33-0268) is also present in AACB mortars.

Cost Analysis

Fig. 13 presents the cost of AACB mortars for different curing conditions. The minimum cost is around 150 Euro/m^3 for the mixture 70FA_10CH_20MG_4M_1SS/SH_0.8 A/B, without the presence of biopolymers and with no temperature curing, while the maximum cost was recorded to be about 350 Euro/m^3 for the mixture composition of 90FA_10CH_8M_2SS/SH_0.8 A/B. An increase in the sodium silicate content and sodium hydroxide concentration is responsible for an increase in the cost of AACB mortars. The minimum cost of mix compositions with the presence of biopolymers was about 200 Euro/m^3 for mixture 70FA_10CH_20MG_4M_1SS/SH_0.8 A/B_0.05CAR, while the maximum cost of the mix composition was detected to be about 400 Euro/m^3 in 90FA_10CH_8M_2SS/SH_0.8 A/B_0.15CAR. Although thermal curing increased the chemical reactions and the compressive strength of AACB mortars, it is also responsible for a relevant cost increase. Fig. 13(b) depicts the costs of mixtures cured with 1 h of heating. The minimum cost was 230 Euro/m^3 for mixture 70FA_10CH_20MG_4M_1SS/SH_0.8 A. However, 260 Euro/m^3 is the minimum cost when 0.05% of the biopolymer carrageenan was used for the same mixture. The costs of mixtures cured with 2 and 3 h of heating are indicated in Figs. 13(c and d). They show an increase for all mixtures but with the same trend already noticed. Fig. 14(a) presents the cost of AACB mortars with different xanthan and for different curing conditions. The minimum cost was recorded to be about 200 Euro/m^3 for mixture 70FA_10CH_20MG_4M_1SS/SH_0.8 A/B with 0.05% xanthan cured at ambient temperature. This cost was about 10% higher than this mix composition without the presence of xanthan. By increasing the curing time, as Fig. 14(c) shows, the cost suffers an increase of around 25%. Fig. 14(d) shows that the minimum cost was about 340 Euro/m^3 for the mix composition of 70FA_10CH_20MG_4M_1SS/SH_0.8 A/B containing 0.05% xanthan and cured with 3 h of heating. Additionally, the maximum cost in the mix compositions containing xanthan powder was obtained to be about 530 Euro/m^3 for the mix composition of 90FA_10CH_8M_2SS/SH_0.8 A/B_0.15XAN cured with 3 h of heating. Abdollahnejad et al. (2015) studied

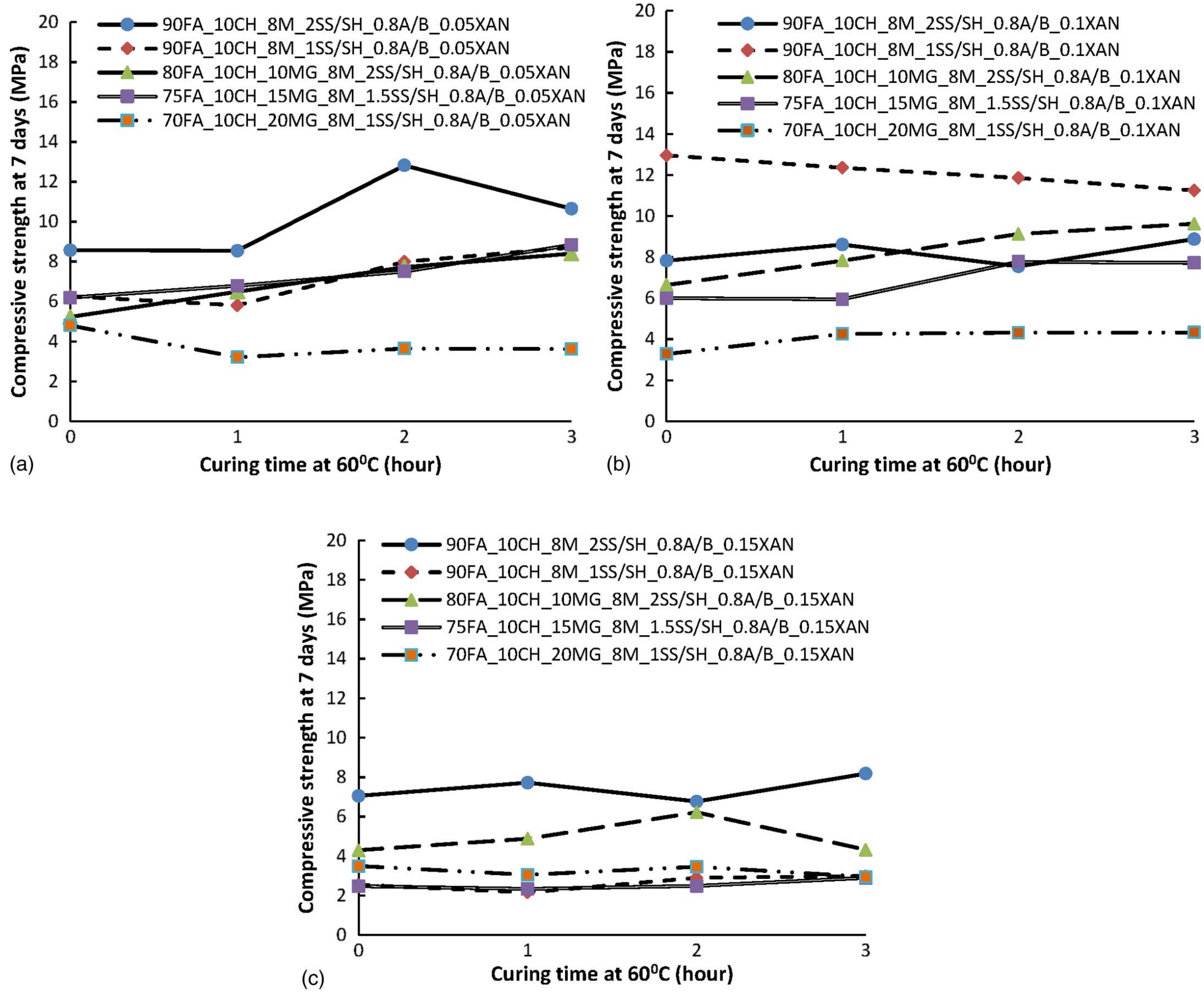
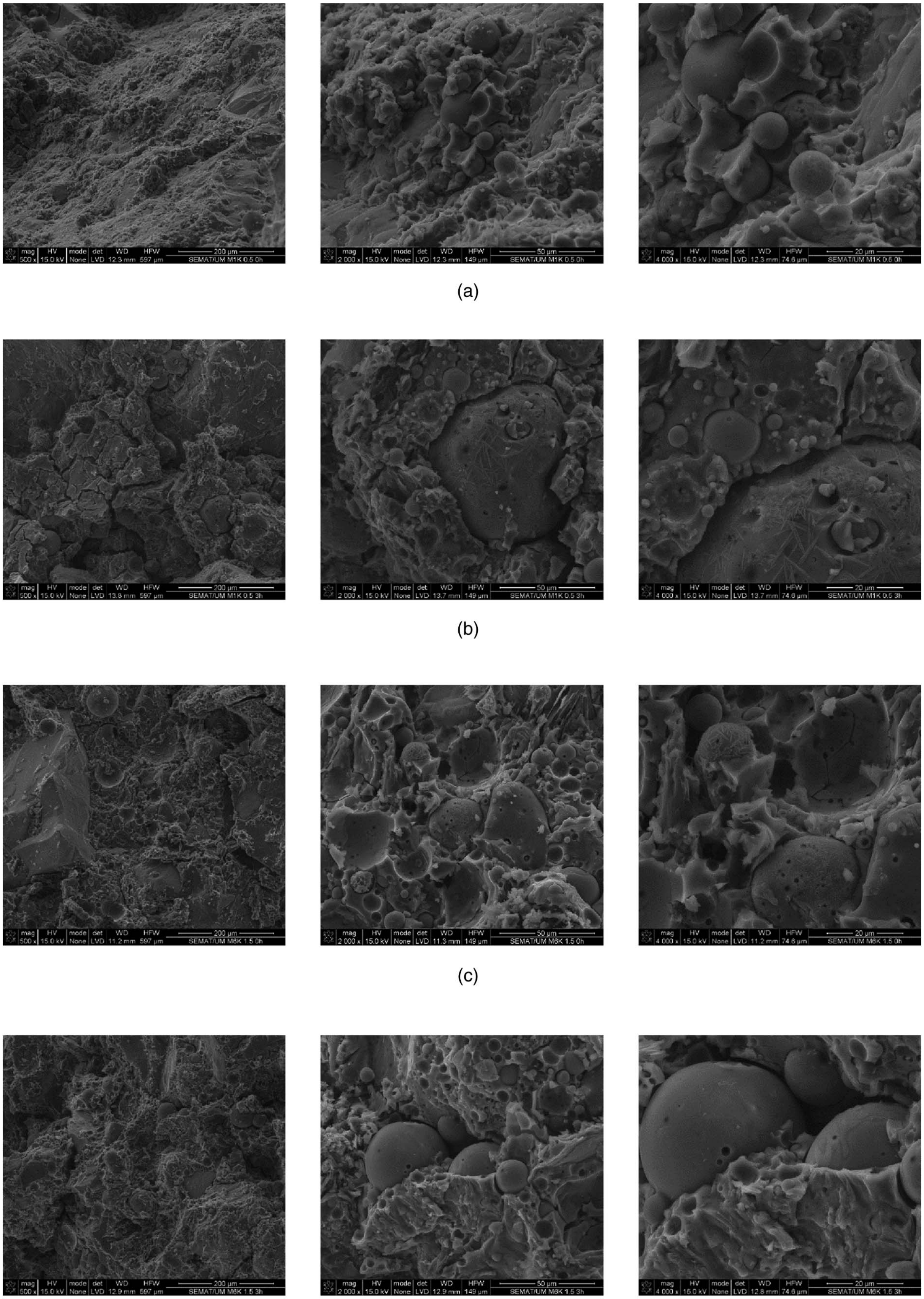


Fig. 10. Compressive strength of AACB mixtures cured at 60°C with 8M and to the following biopolymer xanthan content: (a) 0%; (b) 0.05%; (c) 0.1%; (d) 0.15%

alkali-activated mortars with higher costs between 300 and 600 Euro/m^3. Such high costs were the result of a higher sodium silicate content and a higher sodium hydroxide concentration. However, the same authors found an optimum cost of 67 Euro/m^3 for one-part alkali-activated mortars (Abdollahnejad et al. 2016). This means that AACB mortars still need further investigation in order to become cost-efficient enough.

Conclusions

The results of the present investigations are as follows. The results show that a mixture of 80% fly ash, 10% waste glass, and 10% calcium hydroxide activated with an alkaline activator based on a 4M sodium hydroxide solution show the highest compressive strength. The reduction of the sodium silicate content is associated with a reduction of compressive strength that is slightly compensated by the replacement of fly ash with waste glass. The mixtures containing carrageenan show better workability compared to mix compositions containing xanthan. The results show that an increase in biopolymer carrageenan content is associated with an increase in compressive strength and that the use of 0.1% carrageenan leads to optimum compressive strength. The use of xanthan shows no beneficial effects on the compressive strength of AACB mortars. Several mixtures with xanthan even show a reduction in compressive strength. Thermal curing shows a relevant increase in the compressive strength of AACB mortars. The minimum cost is 160 Euro/m^3 for the mixture 70FA_10CH_20MG_4M_1SS/SH_0.8 A/B, without the presence of biopolymers, while the maximum cost was recorded to be about 350 Euro/m^3 in the mix composition of 90FA_10CH_8M_2SS/SH_0.8 A/B. The use of biopolymers also represents a substantial increase in the cost of the mortars, since the use of the biopolymer carrageenan is associated with the highest cost of around 400 Euro/m^3. Increases in the sodium silicate content and sodium hydroxide concentration are responsible for an increase in the cost of AACB mortars. Curing with temperature also contributes to a relevant increase in the cost of AACB mortars.



(a)

(b)

(c)

(d)

Fig. 11. SEM micrographs of AACB mortars with different curing conditions

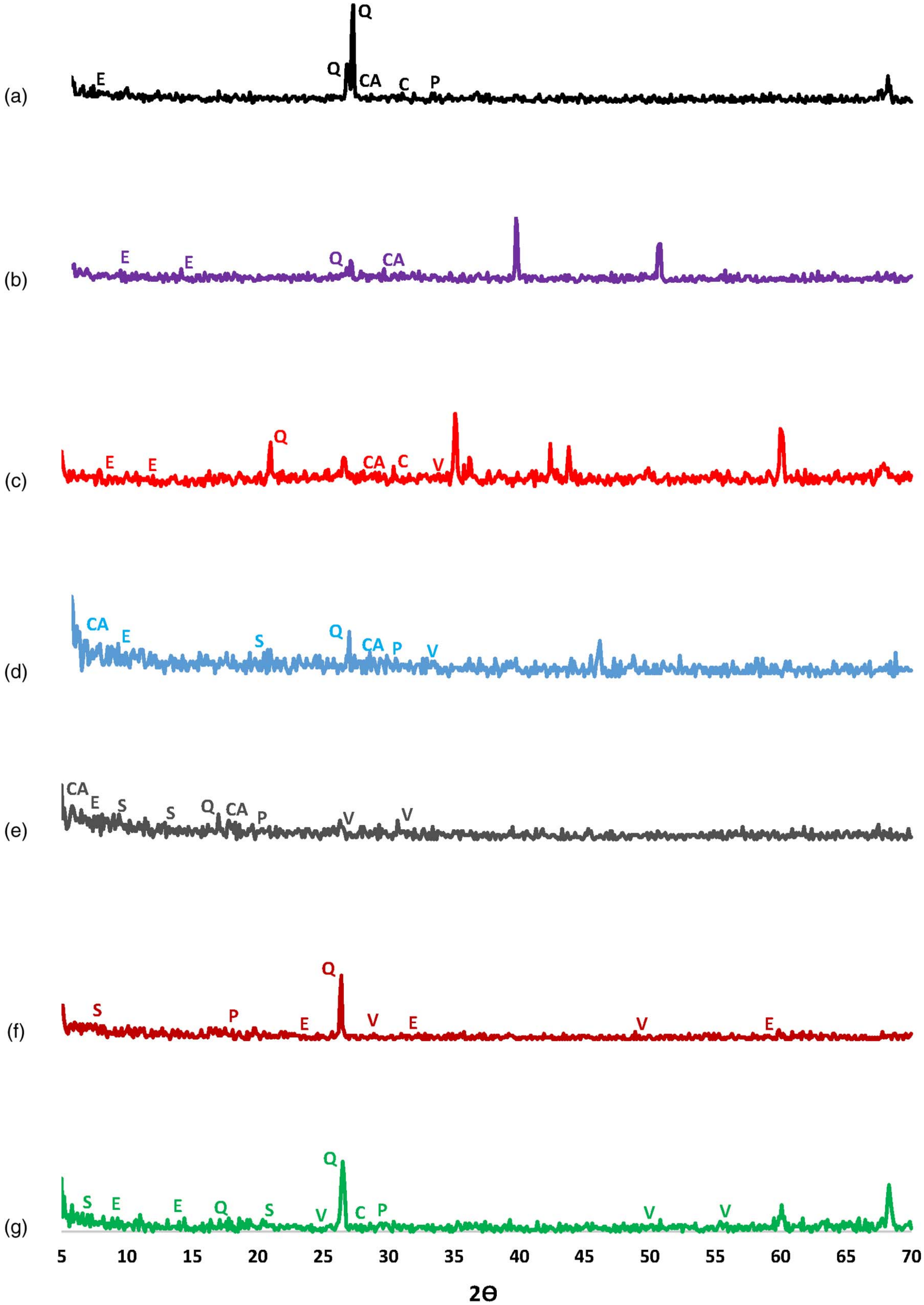
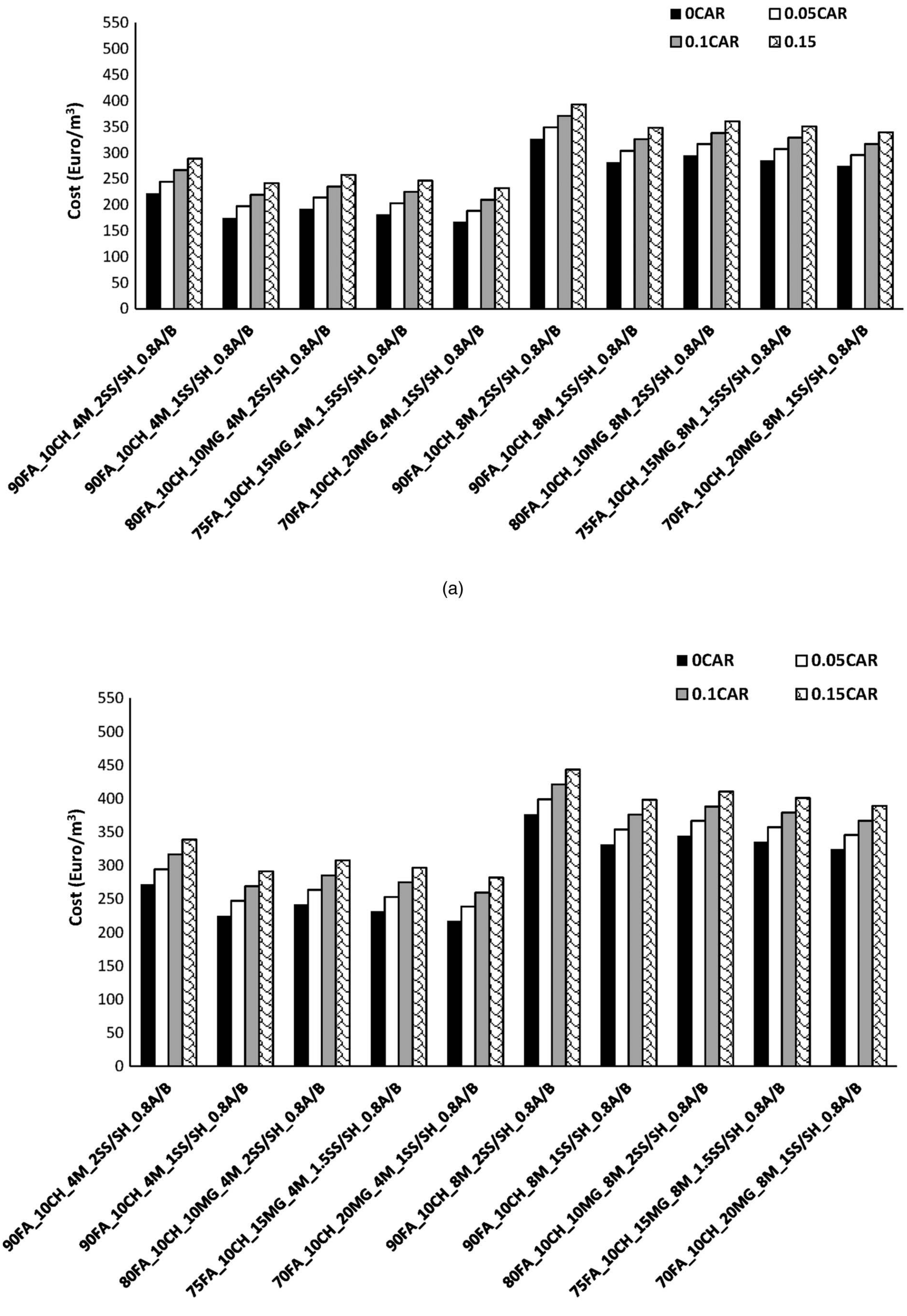


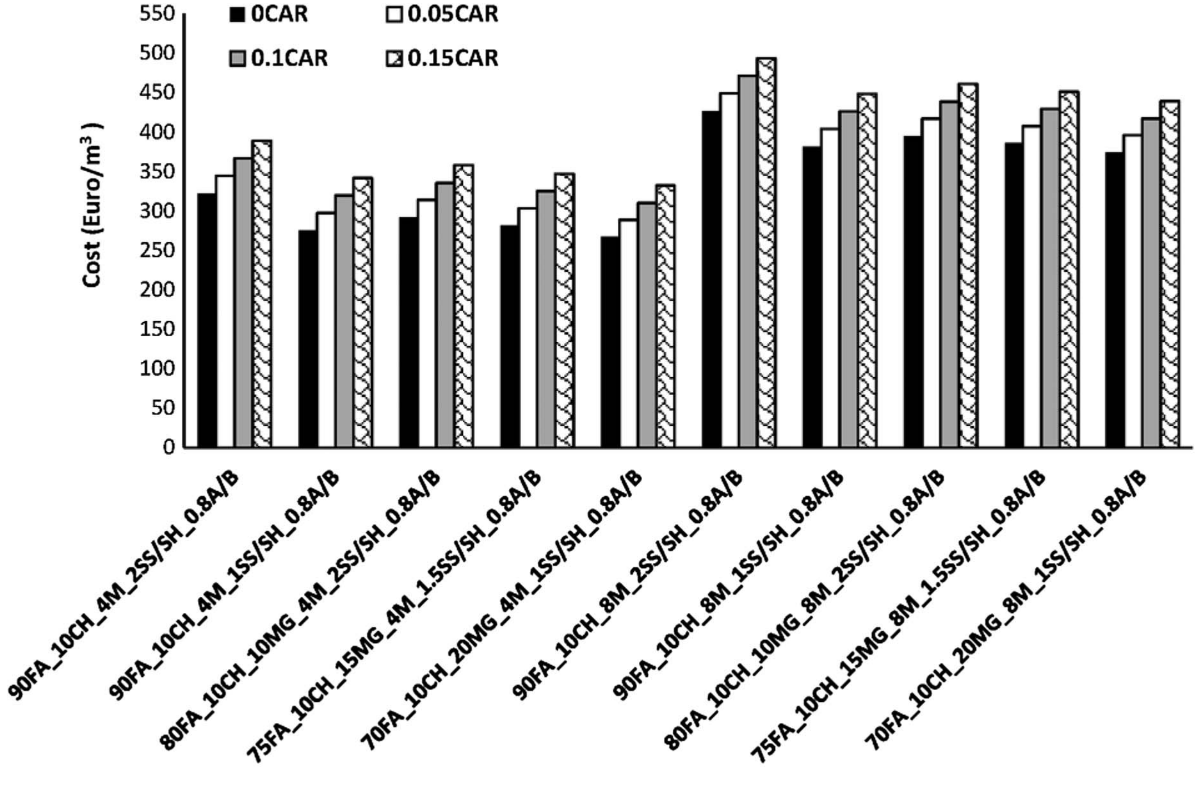
Fig. 12. XRD results for the mix composition of (a) 90FA_10CH_4M_2SS/SH_0.8 A/B_0.15CAR cured at 60°C for 3 h; (b) 90FA_10CH_4M_1SS/SH_0.8 A/B_0.1CAR cured at ambient temperature; (c) 90FA_10CH_4M_1SS/SH_0.8 A/B_0.1CAR cured at 60°C for 2 h; (d) 90FA_10CH_8M_2SS/SH_0.8 A/B_0.05CAR cured at 60°C for 1 h; (e) 90FA_10CH_8M_2SS/SH_0.8 A/B_0.05CAR cured at 60°C for 3 h; (f) 90FA_10CH_8M_2SS/SH_0.8 A/B_0.15CAR cured at ambient temperature; (g) 90FA_10CH_8M_2SS/SH_0.8 A/B_0.15CAR cured at 60°C for 3 h (Q-Quartz, E-Ettringite, P-Portlandite, CA-Calcium Aluminate, C-Calcite, V-Vaterite, S-Stilbite)



(a)

(b)

Fig. 13. Cost of AACB mortars with different carrageenan content and cured (a) at ambient temperature; (b) with 1 h of heating; (c) with 2 h of heating; (d) with 3 h of heating



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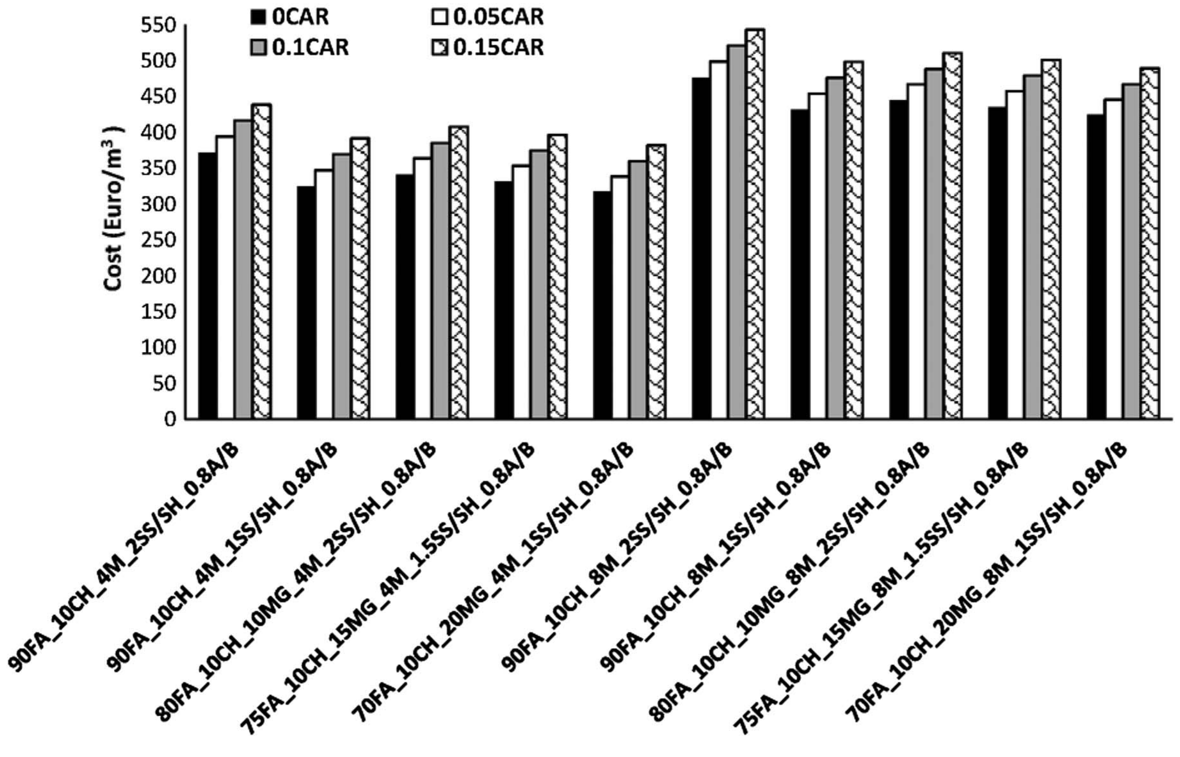
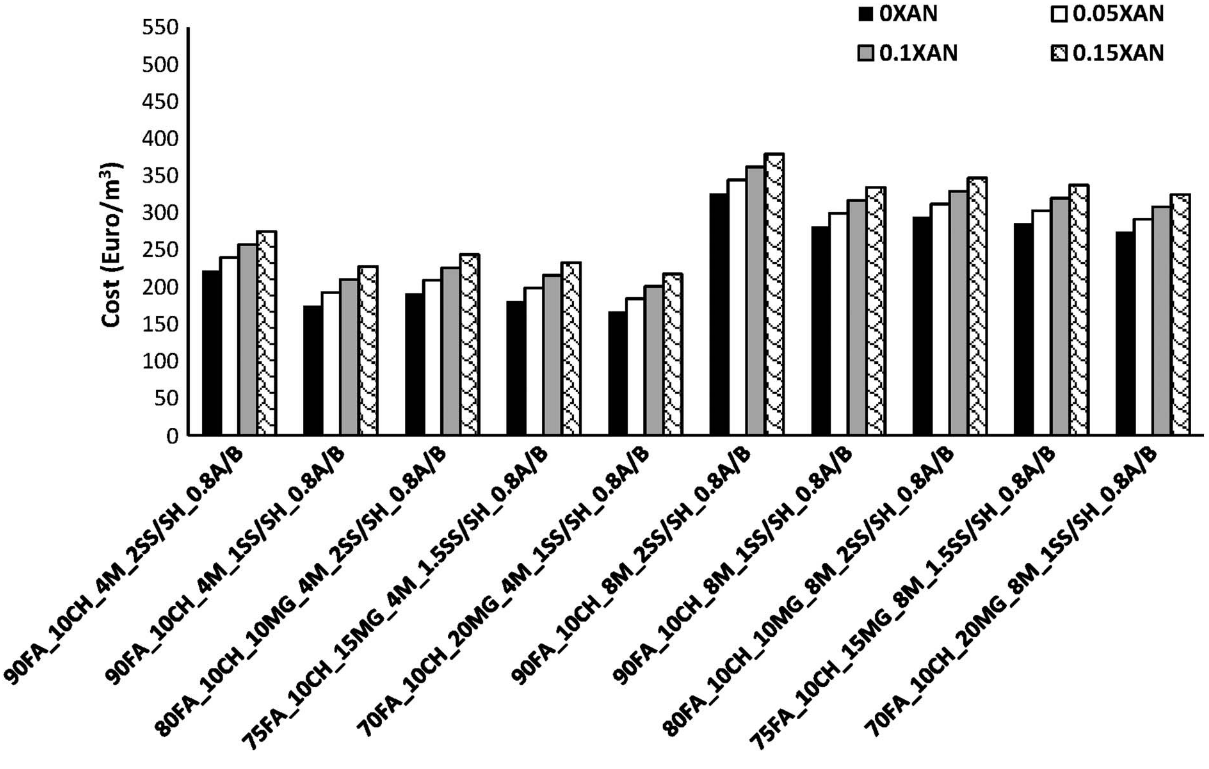
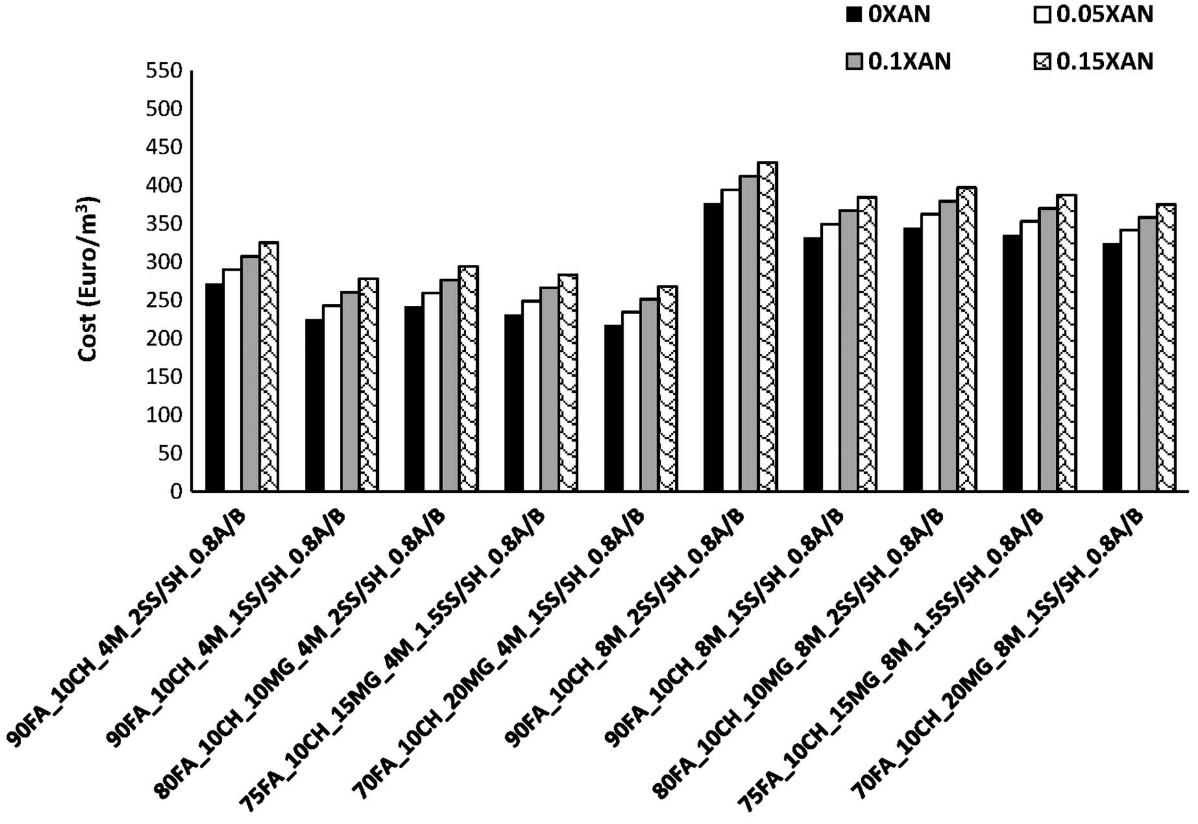


(d)

Fig. 13. (*Continued.*)

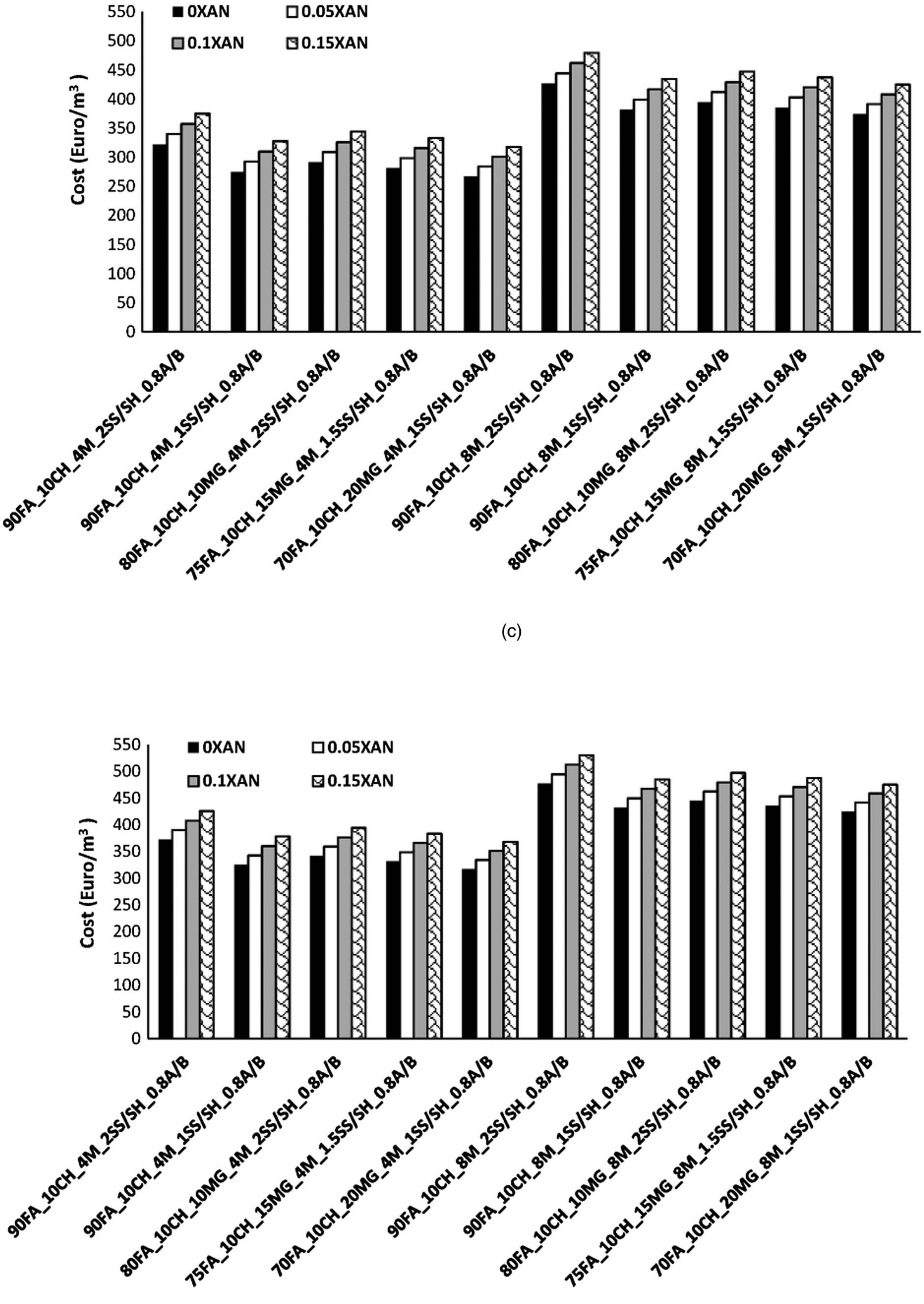


(a)



(b)

Fig. 14. Cost of AACB mortars with different xanthan content and cured: (a) at ambient temperature; (b) with 1 h of heating; (c) with 2 h of heating; (d) with 3 h of heating



(c)

(d)

Fig. 14. (*Continued.*)

Acknowledgments

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