

Influence of vinyl acetate-ethylene copolymer on early-age ettringite formation and behavior in OPC/CAC /hemihydrate gypsum binder system: A case of higher CAC content than OPC content in the binder

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Abstract

In this work, the effect of vinyl acetate-ethylene (VAE) copolymer on ettringite formation and behavior at early age in OPC/calcium aluminate cement/hemihydrate gypsum binder system (BS) was investigated. During the study the VAE dosage was 3 % by weight of the binder. The phase composition of the hydrated BS in air after 6 hours, 1, 3 and 7 days of hardening was investigated by XRD. The microstructure of the hydrated and hardened BS pastes were studied by scanning electron microscopy (SEM). Results from the study indicate that VAE speeds up the hydration of hemihydrate gypsum and slows down the ettringite formation. It was also observed that after 7 days of hardening, the presence of VAE in the BS appears to stabilize the ettringite. There is a change in the appearance of ettringite crystals in VAE-modified BS pastes, i.e. prismatic crystals turn into needle-like crystals, while the average crystals size decreases.

Key words: Cement; Ettringite; Vinyl acetate-ethylene; X-Ray Diffraction; SEM

INTRODUCTION

Dry mix mortars are multicomponent systems that consist of mineral binders (calcium sulfates or ordinary Portland cement), aggregates and additives such as superplasticizers, organic binders, retarders, accelerators, defoamers, cellulose ethers etc [1, 2].

The use of mineral binders regulates the setting time and strength development in mortars. For instance, in some self-levelling underlayments (SLU) or repair mortars fast setting and development of strength is required. To achieve this, calcium alumina cement (CAC) is used to supplement ordinary Portland cement (OPC). The major mineral phase in CAC is monocalcium aluminate (CA), which is very reactive when it gets in contact with water. The minor phases of CAC, which are less reactive, include CA₂ and C₁₂A₇ [2, 3]. The enhanced early ettringite formation leads to an accelerated hydration in OPC/CAC mixtures [2, 4-6].

The mortars in which shrinkage is compensated contain not only OPC and CAC, but also calcium sulfate binders such as anhydrite and hemihydrate gypsum. This kind of sulfate has an influence on the products formed during CAC hydration

[7]. The use of anhydrite leads to formation of large quantities of ettringite during hydration. Ettringite takes in 48.9 wt.% of water into its crystal structure leading to high consumption of water and consequently fast setting of mortars as a result of chemical desiccation [2]. The huge water uptake and development of ettringite needles result in expansion thereby facilitating shrinkage-compensation [2, 7].

In ternary binder systems made up of OPC/CAC and calcium sulfates, additives such as Seignette salt (K/Na tartrate) and lithium carbonate (Li₂CO₃) are included in order to control hydration kinetics so as to achieve a sufficient enough workability period and to provide high early strength [8-10].

Goetz-Neunhoeffer [11] came up with a model that explains the working mechanism of Li₂CO₃ and tartaric acid in CAC/hemihydrate mixtures. He found out that Li₂CO₃ selectively speeds up the hydration of CA and it does so by increasing its rate of dissolution and through formation of a lithium aluminum-layered double hydroxide compound. According to the model, in absence of Li⁺, a non-crystalline layer of aluminium hydroxo hydrate [Al(OH)_x(H₂O)_y] is formed owing to low Al³⁺ solubility under the slightly alkaline conditions.

The [Al(OH)_x(H₂O)_y] wraps the phases of CA and therefore acts as a diffusion barrier hence prevents it from being dissolved further. The use of Li₂CO₃ facilitates the conversion of Al³⁺ into lithium aluminum hydroxide hydrate (LA₂H₁₀), which is precursor for ettringite formation. Therefore Li⁺ catalyzes crystallization of ettringite [11].

Latex polymers are important components in most drymix mortars since they enhance adhesion and cohesion of fresh mortar on solid substrates and improve the brittleness and flexural strength of hardened mortar [12-15]. These effects are achieved as a result of formation of latex films in the cement paste [16-19].

The most common latex polymers are copolymers made from vinyl acetate-ethylene (VAE), styrene/acrylates or styrene /butadiene [20]. A number of studies have been done about these materials [12, 21-23]. Additionally, several studies have been done on the effect of latex polymers on hydration of OPC [22, 24-28]. Recently, Baueregger, et al. [2] conducted a study on the effect of latex polymers on hydration in the OPC/CAC/anhydrite binder system (BS).

For the film to be formed in a cementitious matrix, the latex must undergo particle coalescence, a process that takes place in four main stages [16, 29, 30]. At stage one, the latex is present in an aqueous dispersion. Then the latex forms a dense particle parking (stage two) until the latex particles begin to deform and arrange in hexagonal shape (stage three). Lastly, the polymers undergo diffusion across primary latex boundaries and the particles coalesce into coherent polymer films.

Some researchers believe that only physical interactions take place between cement and polymeric particles [31-33] while others claim that in addition to physical interactions, chemical interactions also occur between cement and polymeric particles [22-24, 34, 35].

Most researchers believe that VAE particles prolong the induction period and reduce rate of cement reaction in the acceleration period. The retardation effect of the polymer is due to the chemical interaction between the polymer and Ca^{2+} ions, leading to formation of calcium acetate [22, 24, 27, 31, 34-36] thereby reducing the CH precipitation [22, 35]. The physical interaction occurs as a result of adsorption of VAE particles onto the surface of the anhydrous and hydrated cement [34].

In this study, vinyl acetate - ethylene copolymer whose chemical structure is shown in **Fig.1.** was used as the BS-modifier.

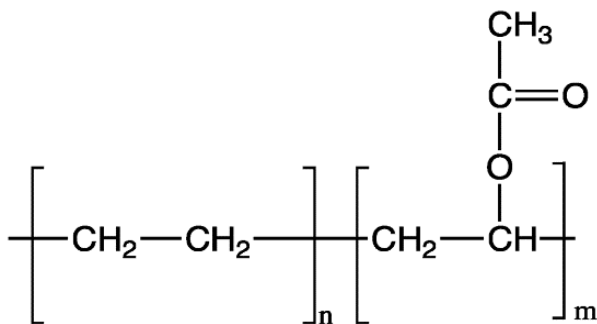


Figure 1. Chemical structure of vinyl acetate-ethylene copolymer

The purpose of this study was to investigate the influence of vinyl acetate-ethylene copolymer on ettringite formation and behavior in an OPC/CAC/hemihydrate gypsum BS at early age (6h to 7 days). The influence of VAE was investigated using X-ray diffraction (XRD) and scanning electron microscopy (SEM)

MATERIAL AND METHODS

Materials:-

The OPC used during the study was CEM II 52,5N from AfriSam, South Africa and its chemical composition is presented in **Table 1.**

The CAC used in this study was ISTRA 40 with approximately 40 wt. % Al_2O_3 . The main mineral phase in the

CAC was CA while the minor mineral phases were C_4AF , C_2AS and $C_{12}A_7$. The chemical and physical characteristics of the CAC are presented in **Tables 2 and 3.**

Table 1: Chemical composition of CEM II 52,5N

Formula	wt. %
CaO	63.79
SiO ₂	20.03
Al ₂ O ₃	4.03
Fe ₂ O ₃	2.59
MgO	2.96
K ₂ O	0.71
Na ₂ O	0.10
SO ₃	2.76
TiO ₂	0.24
Mn ₂ O ₃	2.06
Loss on ignition	6.13

Table 2: Chemical composition of ISTRA 40

Formula	wt. %
CaO	36-40
SiO ₂	≤ 6
Al ₂ O ₃	38-42
Fe ₂ O ₃	13-17
MgO	< 1.5
SO ₃	< 0.4

Table 3: Physical properties of ISTRA 40

Property	Units	Value
Initial setting time	hours	1 - 4
Final setting time		Max 120 min after initial setting time
Blaine specific surface area	m ² /kg	~310-370
Sieve residue on 90 µm	%	< 5
Water demand	%	23 ± 2
Compressive strength (6 hr)	MPa	> 30
Compressive strength (1 day)	MPa	> 50
Bulk density	g/cm ³	~1.15
Specific gravity	g/cm ³	3.2-3.3
Refractoriness	°C	~ 1270

Table 4: Composition of the binder system

Component	Content [wt.%]
OPC	39.14
CAC	45.45
Hemihydrate gypsum G-16 (CaSO ₄ 0.5H ₂ O)	12.63
Antifoam agent 801	0.30
Plasticizer Melment F 10	1.77
Cellulose Ether Tylose H 300 YP2	0.30
Tartaric acid (setting time retarder)	0.20
Li ₂ CO ₃ (accelerator)	0.20
Total	100.00

Table 5: Characteristics of VAE (Vinnapas® RE 5011 L)

Property	Value and unit
Solids content	98-100 %
Ash content	9-13 %
Apparent density	500-600 g/l
Particle size, over 400 microns	< 2 %
Predominant particle size	0.5-8 microns
Minimum film forming temperature	4 °C

Preparation of the binder system and modification:-

The reference BS was prepared by blending OPC, CAC, hemihydrate gypsum and other components shown in **Table 4**. The ratio of OPC to CAC in the BS was approximately 1/1.2. During the preparation of the pastes, a constant water to binder ratio of 0.4 was used. This was done by adding the binder system to water and mixing for about 2 min. The VAE-modified pastes were prepared using a VAE dosage of 3% by weight of the binder. The VAE copolymer (Vinnapas® RE 5011 L) was purchased from Wacker Chemie AG and its characteristics are shown in **Table 5**.

X-ray diffraction:-

The phase composition of the hydrated cement pastes in air after 6 hours, 1, 3 and 7 days of hardening at a temperature of between 21-22 °C and relative humidity $\phi = 60 - 80 \%$ was studied by XRD analysis in an X-ray Diffractometer - DRON-3M, with CuK α radiation, Ni-filter, interval 2 θ (7 – 68) degrees.

Scanning electron microscopy :-

The microstructure of the hydrated and hardened cement pastes were studied by optical microscopy at 300x magnification and by SEM at a magnification of up to 5000x.

RESULTS AND DISCUSSION

XRD analysis:-

The XRD patterns of the BS samples without addition of VAE for different hydration times are shown in **Fig. 2a, b, c** and **d** while the XRD patterns of the BS samples with addition of 3 wt. % VAE are shown in **Fig. 3 a, b, c** and **d**.

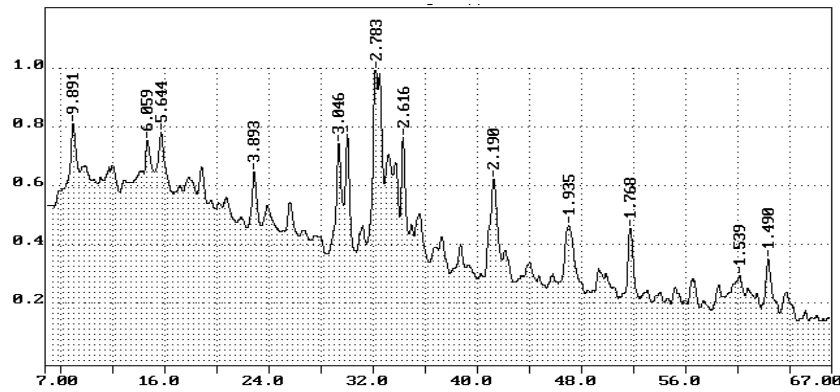


Figure 2a. XRD patterns of the BS sample without addition of VAE after 6 hours of hydration

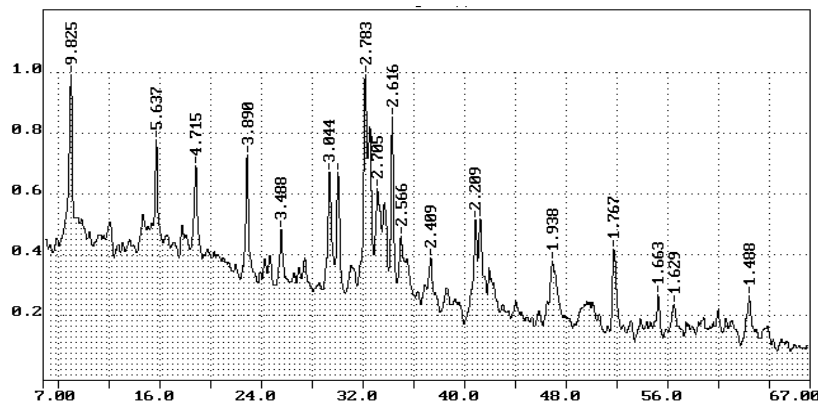


Figure 2b. XRD patterns of the BS sample without addition of VAE after 1 day of hardening

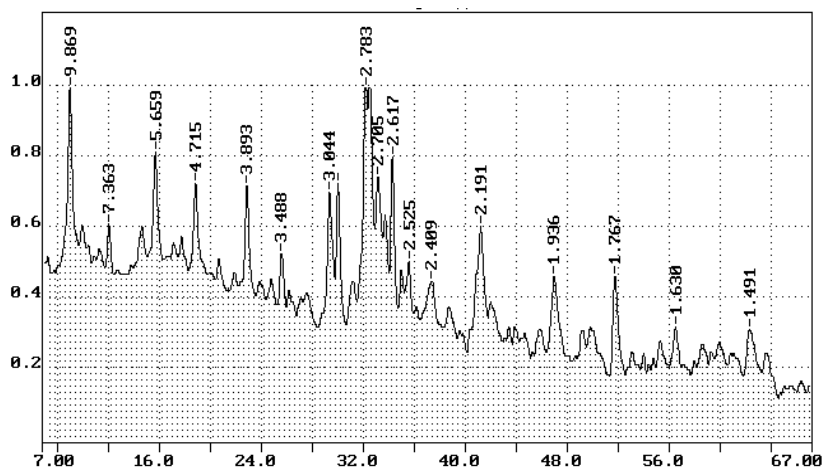


Figure 2c. XRD patterns of the BS sample without addition of VAE after 3 days of hardening

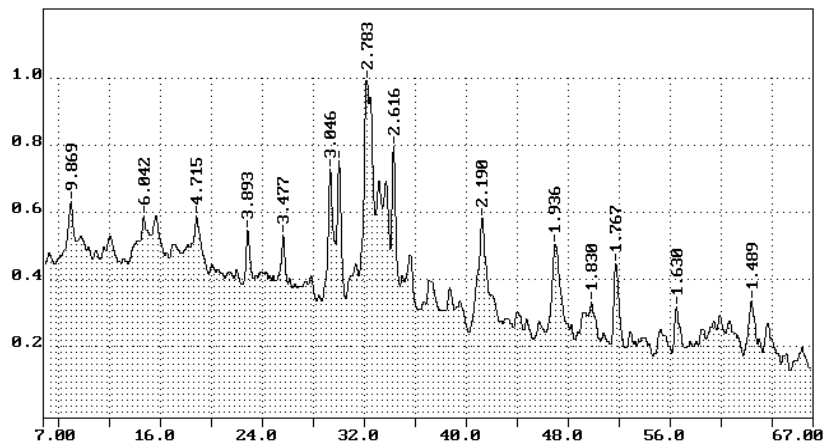


Figure 2d. XRD patterns of the BS sample without addition of VAE after 7 days of hardening

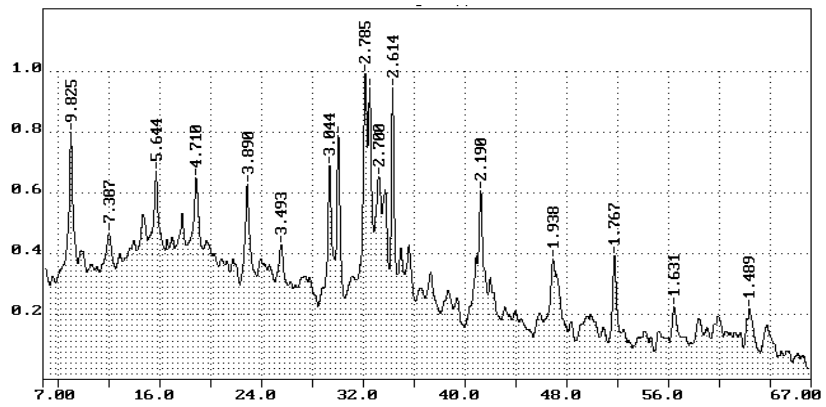


Figure 3a. XRD patterns of the BS sample with 3 wt. % VAE after 6 hours of hydration

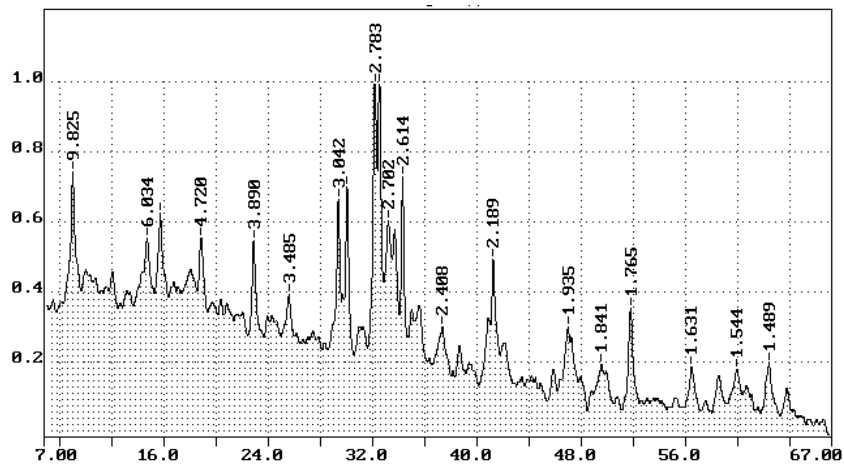


Figure 3b. XRD patterns of the BS sample with 3 wt. % VAE after 1 day of hardening

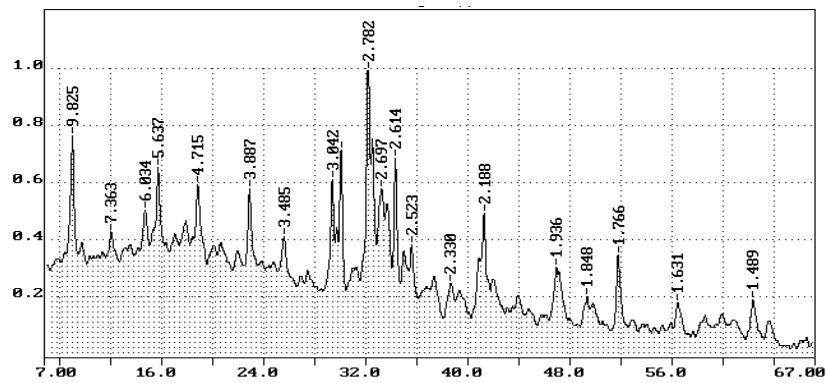


Figure 3c. XRD patterns of the BS sample with 3 wt. % VAE after 3 days of hardening

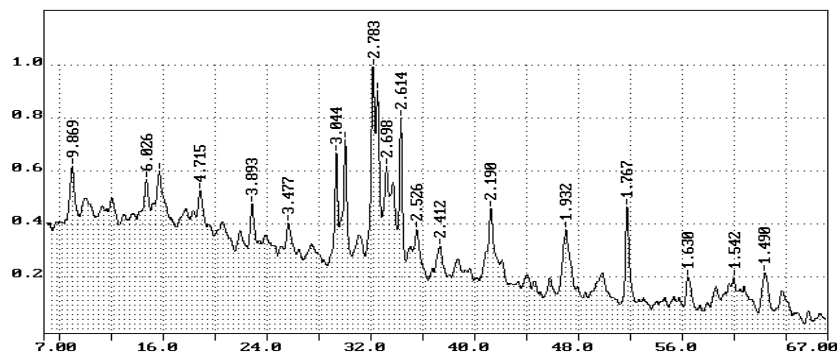


Figure 3d. XRD patterns of the BS sample with 3 wt. % VAE after 7 days of hardening

The XRD analysis shows the peaks corresponding to the main minerals of: Portland cement ($3\text{CaO}\cdot\text{SiO}_2$, $2\text{CaO}\cdot\text{SiO}_2$, $3\text{CaO}\cdot\text{Al}_2\text{O}_3$, $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$), calcium aluminate cement ($\text{CaO}\cdot\text{Al}_2\text{O}_3$, $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$), hemihydrate gypsum ($\text{CaSO}_4\cdot 0,5\text{H}_2\text{O}$) and gypsum ($\text{CaSO}_4\cdot 2\text{H}_2\text{O}$)

From the XRD patterns in **Fig. 2a**, in absence of VAE in the BS, after 6 hours of hydration, intensive peaks corresponding to hydration phases of ettringite $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$ ($d = 9,891$; $5,644$; $3,893$; $2,190\text{Å}$), $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ ($d = 7,651$; $4,283$; $3,046$; $2,616\text{Å}$) and $\text{Ca}(\text{OH})_2$ ($d = 2,616$; $1,935$; $1,768\text{Å}$) can be seen. Along with these, peaks corresponding to $\text{CaSO}_4\cdot 0,5\text{H}_2\text{O}$ ($d = 6,059$; $1,935\text{Å}$) can also be seen. After 1 day of hydration, the intensity of ettringite and gypsum peaks significantly increases (**Fig. 2b**). The peaks corresponding to calcium hydroaluminate $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 13\text{H}_2\text{O}$ ($d = 3,890$; $2,783$; $2,705$; $1,663\text{Å}$) also start to appear. After 3 days of hardening, these trends are persistent (**Fig. 2c**), but on the seventh day of hardening, the intensity of ettringite peaks decreases while that of gypsum and Calcium hydroaluminate increases (**Fig. 2d**). However, even after 7 days of hardening, there is presence of a fairly large fixed amount of ettringite in the hardened binder.

Fig. 3a indicates that the use of 3 wt. % VAE in the BS does not reduce the rate of ettringite formation after 6 hours of hydration. However after 1, 3 and 7 days of hardening, the intensity of the ettringite peak at $d = 9,89\text{Å}$ in the case of

VAE-modified BS is much lower than in the BS without VAE addition (**Fig. 3b**, **Fig. 3c** and **Fig. 3d**).

Therefore, it is evident that addition of VAE in the BS slows down the process of ettringite formation. In absence of VAE in the BS, the amount of ettringite decreases with hydration time. This is because, in the conditions of inadequate water, VAE-absent cements undergo decomposition leading to the formation of gypsum $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ and $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 13\text{H}_2\text{O}$. Ettringite is stabilized with addition of VAE in BSs and in the conditions of inadequate moisture, its decomposition into $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ and $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 13\text{H}_2\text{O}$ is slowed down.

SEM analysis :-

The microstructures of the BS samples in absence of VAE after: 1 day of hydration and 7 days of hardening are shown in **Fig. 4a₁**, **a₂** and **Fig. 4b₁**, **b₂** respectively while **Fig. 5a₁**, **a₂** and **Fig. 5 b₁**, **b₂** show the microstructures of the BS samples with 3 wt. % VAE addition after: 1 day of hydration and 7 days of hardening respectively.

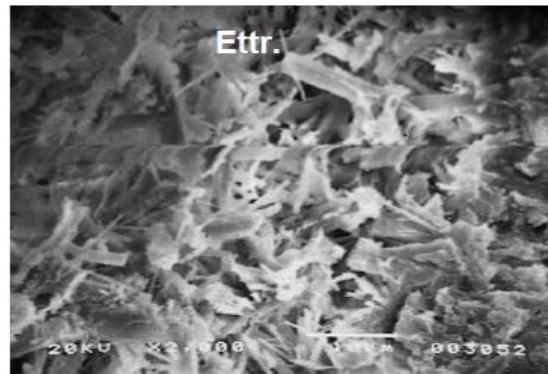


Figure 4a1. SEM micrograph of the BS sample without VAE addition after 1 day of hydration

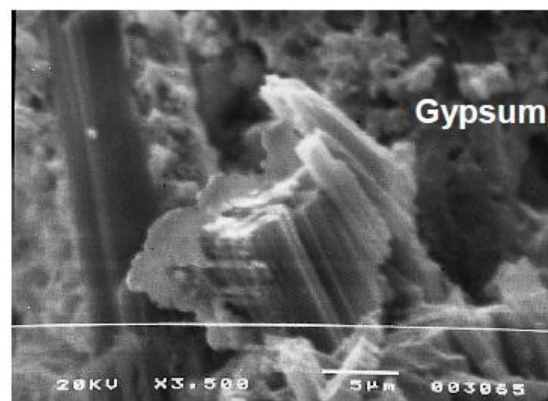


Figure 4a2. SEM micrograph of the BS sample without VAE addition after 1 day of hydration

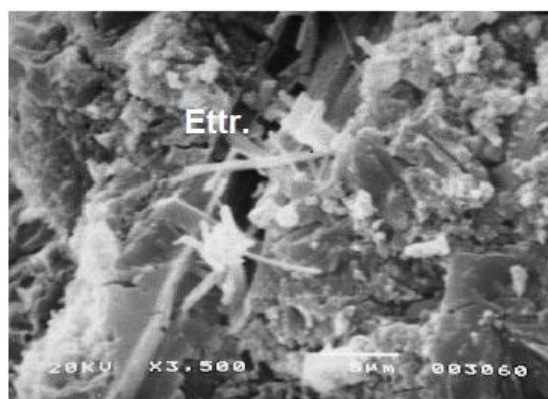


Figure 4b1. SEM micrograph of the BS sample without VAE addition after 7 days of hardening

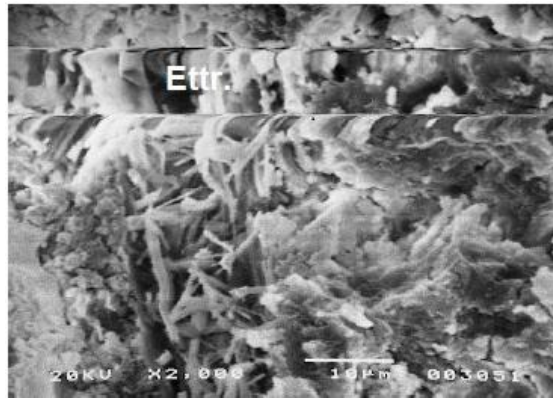


Figure 4b2. SEM micrograph of the BS sample without VAE addition after 7 days of hardening

From **Fig.4a1** and **Fig.4a2**, after 1 day of hydration, significant amount of ettringite and gypsum crystals can be seen. After the 7 days of hardening, as seen in **Fig.4b1** and **Fig.4b2**, the amount of ettringite crystals slightly reduces. Large lamellar

crystals formation can be seen, which probably are crystals of calcium hydroaluminate or calcium monohydro sulfoaluminate.

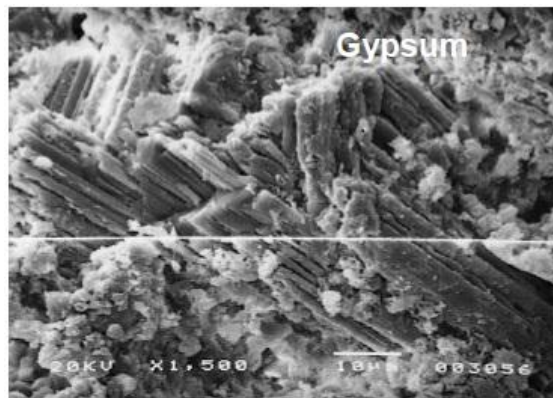


Figure 5a1. SEM micrograph of the BS sample with addition of 3 wt. % VAE after 1 day of hydration

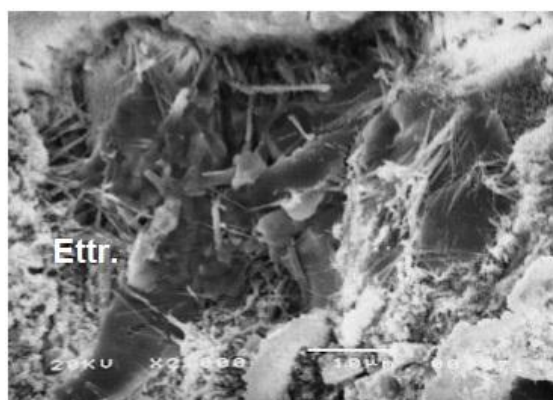


Figure 5a2. SEM micrograph of the BS sample with addition of 3 wt. % VAE after 1 day of hydration



Figure 5b1. SEM micrograph of the BS sample with addition of 3 wt. % VAE after 7 days of hardening

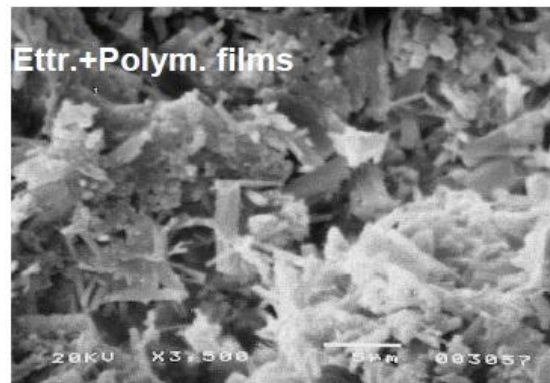


Figure 5b2. SEM micrograph of the BS sample with addition of 3 wt. % VAE after 7 days of hardening

With addition of 3 wt. % VAE in the BS as seen in **Fig.5a1** and **Fig.5a2**, after 1 day of hydration, large prismatic gypsum crystals are formed. The amount of ettringite is relatively small and ettringite crystals, which are needle-like in shape, are smaller in size than those observed in VAE-absent BS. From **Fig.5b1** and **Fig.5b2**, after 7 days of hardening, gypsum crystals are still present and the amount as well as the size of ettringite crystals increases.

Thus, the study of the BS microstructure using SEM method confirms that addition of VAE in the BS slows down the formation of ettringite at the early stages of hydration. In VAE-modified BS, ettringite crystallizes in form of tiny needle-like crystal forms. Moreover, VAE addition in the BS stabilizes the ettringite formed in the early stages of hydration.

CONCLUSION

Ternary binder systems consisting of Portland cement, calcium aluminate cement and hemihydrate gypsum are used for applications such as self-leveling flooring or repair mortars, which require a fast setting and low shrinkage. VAE use in the BS enhances flow and self-levelling properties. The motivation behind this study was to understand the influence of addition of VAE into the BS on ettringite formation and behavior at early age (6h to 7 days).

This study demonstrates that modification of the BS with VAE slows down the formation and crystallization ettringite, one of the main crystal hydrates of cement hydration. The use of VAE also prevents the formation of ettringite crystals in liquid phase. This is probably due to the decrease in degree of liquid phase saturation by ions of Ca^{2+} , OH^- , SO_4^{2-} etc. as a result of reduction in solubility of initial cement in water or change in liquid phase properties such as viscosity.

The reason for the slow formation of ettringite is probably due to the fact that VAE prolongs the induction period and has a retardation effect on the cement reaction rate during the acceleration period. The chemical explanation for this is that during hydration, vinyl acetate in VAE undergoes hydrolysis to produce the acetate ion (CH_3COO^-), which then interacts with Ca^{2+} , leading to formation of calcium acetate. This reaction consumes Ca^{2+} thereby creating unreacted sulfate ions which further delayed the hydration process and consequently delayed the formation of ettringite. The physical explanation for the retardation is that the polymeric particles get adsorbed on the surface of cement during the acceleration period.

In VAE-modified BS pastes, the appearance of ettringite crystals changed from prismatic to needle-like crystals, while the average crystals size decreases. A polymeric film deposit formation on the surface hydrated cement phases in the VAE-modified BS pastes was observed. Additionally, VAE stabilizes the formed ettringite and therefore prevents its

destruction and recrystallization into calcium monosulfoaluminate hydrate or calcium hydroaluminate and gypsum when there is water deficiency in the hydration system.

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