Vietnam Journal of Science and Technology ***62*** *(2024)*
doi:10.15625/2525-2518/18543

Waste solution of acrylic-based emulsion with low solid content for cement concrete curing: a lab-scale study

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Received: 18 July 2023; Accepted for publication: 8 July 2024

Abstract. In this study, we investigate using acrylic and acrylic-styrene emulsions as curing solution to enhance water resistance and prevent cracking in cement concrete surfaces. Our main objective is to investigate the possibilities of utilizing washing waste generated during the Rhomn & Haas factory - Dow Chemical Vietnam production process. This waste is currently being treated as solid even though it contains anywhere from 10 – 45 % solid content. Through laboratory-scale experiments and analysis, we aim to assess the feasibility of employing this waste material as a cost-effective, technically sound, and environmentally sustainable solution for curing compound of concrete. The findings of this research have the potential to contribute to the advancement of eco-friendly waste management techniques while improving the durability and effectiveness of cement structures.

Keywords: acrylic-based emulsion, solid waste, concrete curing, shrinkage crack, durability.

Classification numbers: 2.5.3, 2.9.3, 3.3.2.

1. INTRODUCTION

Moisture curing is crucial for cement concrete materials as it promotes proper hydration and strengthens the material over time. Under normal temperature conditions, cement concrete must be cured for a period of at least 28 days to achieve its final strength development. In addition, in hot weather conditions, it is essential to adequately cure the concrete to prevent macroscopic surface cracking and microscale damage to the microstructure, which can significantly affect the concrete's mechanical strength and lead to long-term indirect effects on its durability, such as cavitation, corrosion, and reduced service life of concrete structures.

While voids and cracks are typical occurrences in cement concrete materials due to the intricate nature of the cement hardening process, completely mitigating the effects of shrinkage during concrete curing can be a formidable challenge. Various factors, including the evaporation of free water, crystallization reactions, and creep load, can all contribute to shrinkage. Moreover, plastic settlement and shrinkage cracking are prevalent issues during the curing process for all types of concrete. Research findings have indicated that in conditions characterized by hot weather, low humidity, and high winds, approximately 80% of the cracks in concrete elements during the early stages of curing can be attributed to the rapid evaporation of free water from the concrete surface [1]. These cracks typically manifest as short diagonal or scattered cracks, as illustrated in Figure 1.

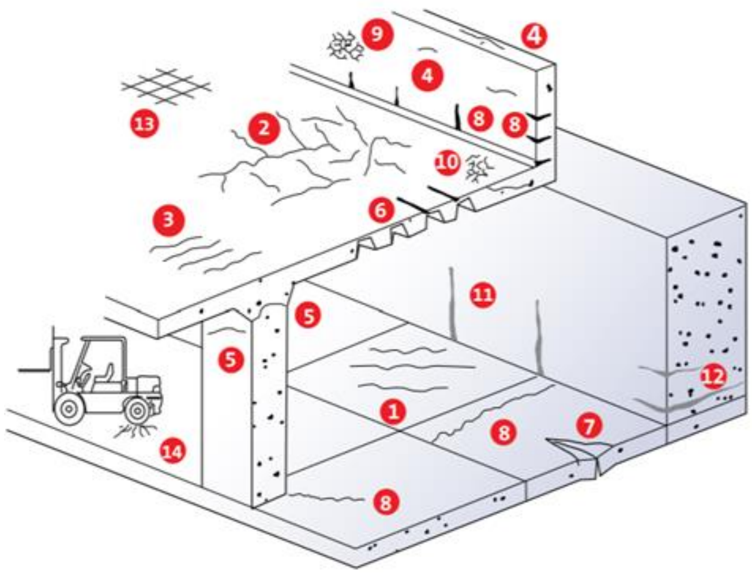


Figure 1. Common types of cracks in cement-concrete structures and their causes. (Plastic settlement: 4, 5, 6, 13; Plastic shrinkage: 1, 2, 3; Early thermal contraction: 11, 12; Long-term drying shrinkage: 8; Crazing: 9, 10; Cracks due to overloading: 14) [2].

There are various methods to minimize cracking caused by plastic shrinkages, such as appropriate curing measures during the hardening process or incorporating fibers (such as steel or polypropylene) into the concrete mix to increase tensile strength and prevent crack formation. Applying thin coating materials to the concrete surface can also help prevent free water evaporation and the formation of cracks. The use of curing solutions that form stretchable films for surface coating is becoming increasingly common on concrete construction sites. One such solution is acrylic resin, formed by polymerizing acrylate monomers and emulsified by surfactant stabilizers, then dispersed and suspended in water as a continuous phase. In term of film forming mechanism, due to its high molecular weight, the coating film is formed on the surface by a physical mechanism through the gradual evaporation of the aqueous solvent. This process occurs in three successive phases: (1) primary dispersion with the evaporation and concentration build-up; (2) spherical packing of particles with deformation caused by capillary force; and (3) rhombic dodecahedral packing with continuous inter-diffusion [3]. Acrylic-based film-forming agents are generally considered inert when applied to cementitious materials, which means they do not undergo chemical reactions with the concrete surface they cover. In previous study, Ito et al., reported that applying the acrylic based emulsion does not hinder hardening and strength development and reduces cracks due to plastic shrinkage [4]. Instead, they form a protective layer on the surface of the concrete, improving its resistance to water, chemicals, and abrasion (Figure 2). In the study conducted by Lazar et al. [5], it was demonstrated that the utilization of pure acrylic film-forming agents can not only improve the properties of concrete but also enhance its visual appearance. This enhancement can result in either a glossy or matte finish, depending on the desired aesthetic effect.

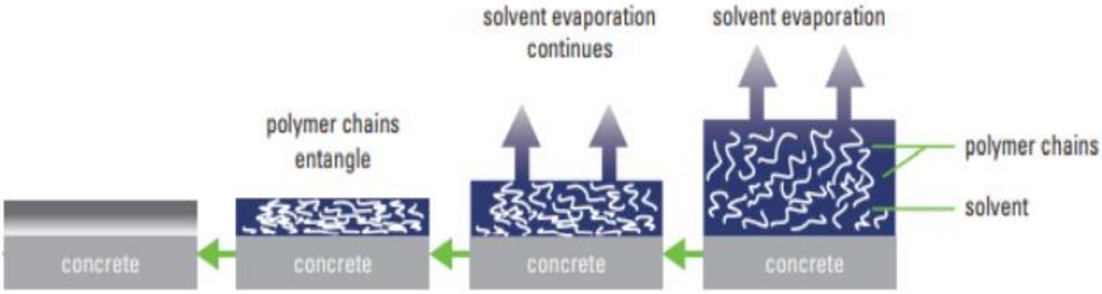


Figure 2. From left ot right: Illustration of solvent evaporation and film-forming process of acrylic-based emulsion on the concrete substrate [6].

This study investigates using acrylic and acrylic-styrene emulsions as coating solutions to prevent cracking and increase water resistance in cement concrete surfaces. Specifically, we examine the use of washing waste from the production process at Rhomn & Haas factory - Dow Chemical Vietnam, which contains a solid content of 10-45% and is currently treated as solid waste. Through experiments in laboratory scale and analysis, we aim to evaluate the feasibility of utilizing this waste material as a cost-effective, technically sound, and environmentally sustainable solution for concrete surface protection.

2. MATERIALS AND EXPERIMENTAL METHODS

2.1. Solid waste of acrylic solution and analysis

Figure 3. Milky white solution of acrylic-based emulsion with different solid contents.

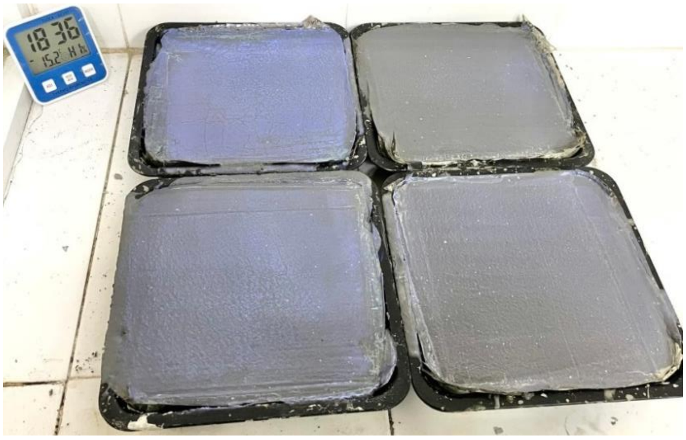
The two emulsified materials, acrylic (AC) and acrylic-styrene (AS), are milky white solutions with a solid content that varies widely due to their nature of washing sink waste: 10.2 %, 34.6 %, and 45.1% for acrylic emulsion, and 10.2 %, 17.5 %, and 30.1 % for acrylic-styrene emulsion. Depending on the solids content, these emulsions are slightly viscous (Figure 3) with density values of 1.05 - 1.12 and a neutral pH of 7 - 9. Therefore, they are readily soluble in water. However, since they are derived from solid waste, the stability of the solution is low. Therefore, after a while in the cup, the emulsions tend to settle in the bottom but can be easily dispersed by gentle shaking. Before utilization, those emulsified solutions were subjected to Fourier-transform infrared spectroscopy (FTIR) analysis, conducted within the wavelength range of 4000–400 cm^{-1} using FT-IR Spectrometer NICOLET 6700, Thermo Fisher Scientific.

2.2. Preparation of mortar samples and experimental methods

We used PC40 cement material (Vicem Ha Tien 1), river sand, fine crushed stone, and water to prepare cement mortar samples. PC40 cement material was chosen as it meets the mechanical and physical criteria standard TCVN 9202:2012. The river sand was washed, dried, and analyzed for particle size composition using sieves (national standard TCVN 7572:2006), which resulted in a finesse modulus of 1.65, indicating that the sand is fine. Crushed stone with grain sizes above/under the 2.5/5 mm sieves was used to add larger grain sizes to the river sand. Additionally, the remaining dust in crushed stone ensures that the surface of the concrete casting is smooth and glossy while making scattered cracks more easily observable. The mixing ratio of river sand to finely crushed rock was fixed at 1.2:1 in this experiment. We conducted experiments using three types of samples (Figure 4) with different objectives.

Round disc-shaped cement paste

Round disc-shaped cement mortar



Thin slab sample of cement mortar

Figure 4. Three types of cement paste/mortar samples for test

The first type was a round disc-shaped cement paste sample (Figure 4-above-left) used to monitor the effect of coating on water retention during hardening or cement curing. For this group, we mixed cement and water in a ratio of 1:0.28 and poured the mixture into plastic Petri dishes with a diameter of 95mm and a thickness of 18mm. We permitted them to air dry for 30 minutes at room temperature before applying a film coating. We sprayed each sample's surface with an emulsion solution (dosage estimated at 300 g/m^2). Subsequently, we positioned these samples inside a controlled chamber with regulated temperature and humidity settings. Over a duration of 4 hours, we continuously monitored their weight to compare the evaporation rates between the coated and uncoated samples. Finally, we plotted the free water evaporation results at 10-minute intervals by calculating the weight difference before and after each time interval.

The second type of sample was a round disc-shaped cement mortar sample (Figure 4-above-right) used to monitor the effect of coating on resistance to water absorption into the hardened mortar. For this group, we mixed cement, sand, and water in a ratio of 1:2.8:0.56 using a planetary mortar mixer. We poured the mixture into a silicon resin mold with a diameter and thickness of 95mm and 18mm, respectively. Following a 28-day curing period, we meticulously smoothed the surface of the hardened mortar using sandpaper to achieve uniformity and minimize imperfections. Subsequently, we coated each mortar substrate with an emulsion solution, applying approximately 2.13 ± 0.2 grams (equivalent to an estimated dosage of 300 g/m^2). These coated substrates were left to air dry for 2 hours at room temperature. After this preparation, we immersed half of the substrate's thickness in a water bath to induce capillary-driven water absorption. Over the course of 72 hours, we regularly weighed these samples to determine the rate of water absorption at 3, 10, 24, 48, and 72 hours. To assess the depth of water infiltration, we employed a hydraulic jack to fracture the mortar samples.

The third type of sample was a small and thin slab sample of cement mortar (Figure 6-below) used to monitor the influence of the coating film on crack reduction due to shrinkage. We mixed cement, sand, and water in a 1:1:0.4 (Sample A) and 1:1:0.45 (Sample B) using a planetary mortar mixer and poured the resulting mixture into a mold with 200mm x 200mm x 20mm. After allowing the surface to dry for an hour at room temperature, we sprayed each sample with emulsion solution acrylic AC and acrylic-styrene AS (dosage = 300 g/m^2 as estimated). Then, we placed them in a controlled chamber for 12 hours to evaluate the effectiveness of the emulsion coating in reducing the free water evaporation rate and preventing shrinkage cracking on the surface of the cement mortar. We maintained a steady temperature of 20 ± 2°C and relative humidity of 43.8 ± 6.8% to encourage free water vapor evaporation and the potential formation of cracks. Additionally, we captured photos to evaluate the effectiveness of the various emulsion coatings.

3. RESULTS AND DISCUSSION

3.1. Result of analysis of waste solution of acrylic

The FTIR infrared spectroscopy analysis of AC and AS emulsions is shown in Figure 5. The spectra show similarities in the main links, including a broad and deep peak band at wave number #3420 cm^{-1}, which characterizes the highly polar O-H bond of the solvent, and a wave number at #1730 cm^{-1} for the C=O bond of the COO- radical (carboxyl group). Additionally, wave numbers #1183 - 1294 cm^{-1} show bond elongation of the alkyl group (C-O) in acrylic acid. The main difference between acrylic and acrylic-styrene emulsions can be seen in the wave numbers #750 cm^{-1} and #1450 cm^{-1}, characteristic of C-H binding and C=C stretching, respectively [7].

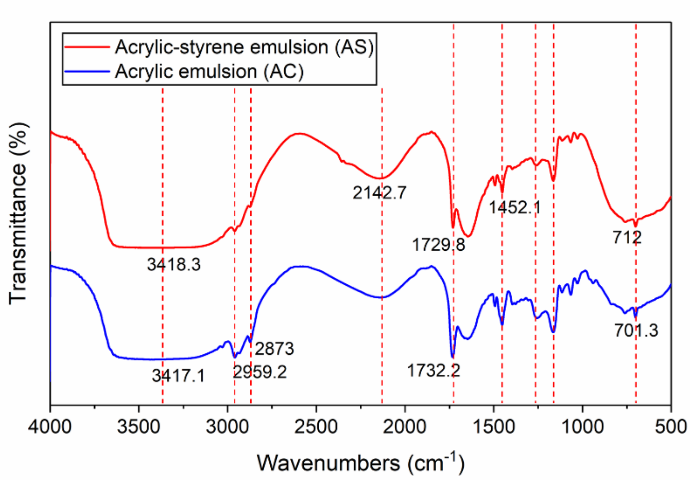


Figure 5. FTIR spectrum of acrylic emulsion (AC), acrylic-styrene emulsion (AS).

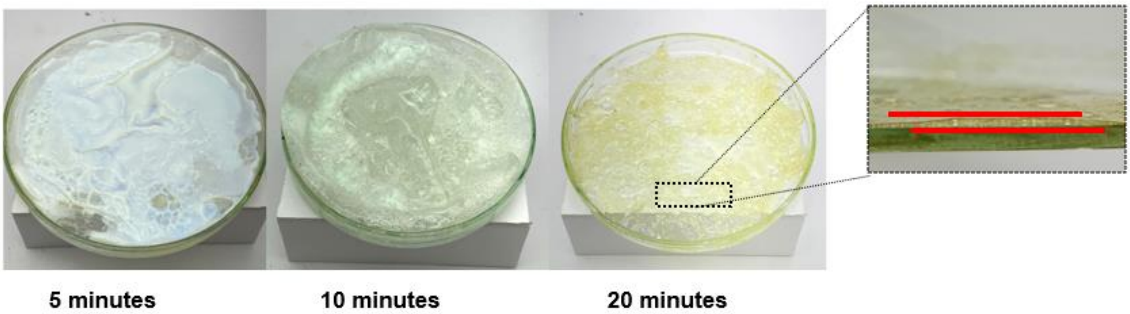


Figure 6. Acrylic emulsion after drying 5, 10 and 20 minutes at 120 ^{0}C in a petri dish.

From the observations made in Figure 6, it is clear that the drying and thickening of the acrylic resin solids due to solvent evaporation align with the film-forming mechanism discussed earlier. During the initial drying stage (5 minutes at 120^{0}C), water evaporation occurs rapidly at the emulsion's surface, which is directly affected by the heat source. This leads to the formation of a surface film that retains water inside. After 10 minutes of drying, the surface film significantly reduces the surface area available for evaporation, causing water evaporation to slow down and occur through diffusion. Gas molecules then penetrate inside and replace the lost volume of water, creating a repulsive force that causes the membrane to swell. After 20 minutes of drying, all the solvent has evaporated, resulting in a colloidal solid. This solid appears darkened on the glass dish due to slight combustion (Figure 6).

3.2. Result of water retention of cement paste samples

The surface of the cement paste is depicted visually in Figure 7-left, showcasing the coating film that has been formed. While the film adheres well to the surface, some defects are apparent, which may be attributed to a low dispersion of polymer particles or excessive emulsion solids. Locations with fewer polymer particles are challenging to pull, leading to the formation of defects as the solvent evaporates. Conversely, areas of high polymer particle concentration can result in condensation. Additionally, uneven surfaces of the cement substrate or the emulsion's separation into the water on the surface may also contribute to the formation of defects. The transparent coating enables a clear view of the substrate, with no visible connections between the cement paste and the coating material.

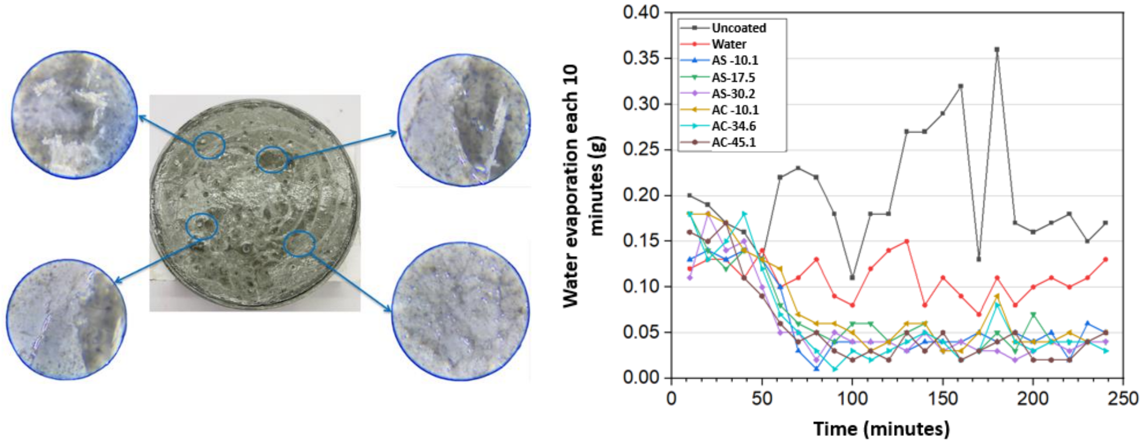


Figure 7. (left) Visualisation of coating film on the surface of cement paste; (right) Water evaporation rate each 10-minutes intevall during the drying process (note: AS = acrylic styrene; AC = acrylic).

In Figure 7-right, the weight of water evaporated every 10 minutes is plotted, demonstrating the water retention effect of the coating film compared to the uncoated and water-sprayed samples. The film exhibited a moisturizing effect within the cement paste, reducing water evaporation. Within the first 90 minutes after casting cement paste in the mold, there was no significant difference in water retention as the aqueous solvent in the emulsion evaporated along with the water within the cement paste. However, after 90 minutes, when the film dried on the surface, the amount of water evaporated was significantly reduced. The moisturizing effect was more significant for emulsions with higher solid content, as the lower solvent content led to faster surface drying and earlier formation of a more uniform and robust film. Increasing the solids content also increased the polymer particle content per volume, resulting in a stronger and more uniform film with greater thickness.

3.3. Result of water absorption of mortar samples

The qualitative and quantitative observations provide insights into the water resistance properties of the mortar samples. The qualitative experiments show that the thickness of water permeation through the contact surface of coated and uncoated disk-shaped samples differs significantly. Moreover, the thickness values remain uniform and stable across all positions on each sample's contact surface. The coated samples display better impermeability than their uncoated counterparts under the same experimental conditions. The photographs of the formed film layer demonstrate that the sprayed emulsion achieves excellent water resistance properties, with a thick film layer and a smooth surface exhibiting the lotus leaf effect (Figure 8). These results highlight the crucial role of addressing surface defects in ensuring water resistance in mortar surfaces.

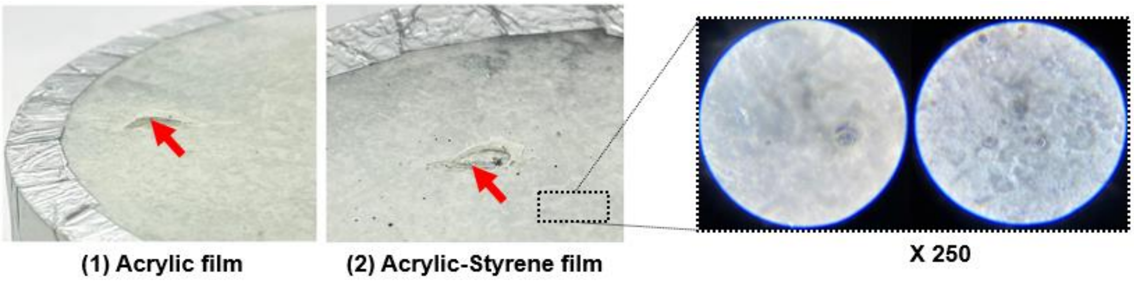


Figure 8. Formed film layer on the cement mortar substrate due to sprayed emulsion of acrylic

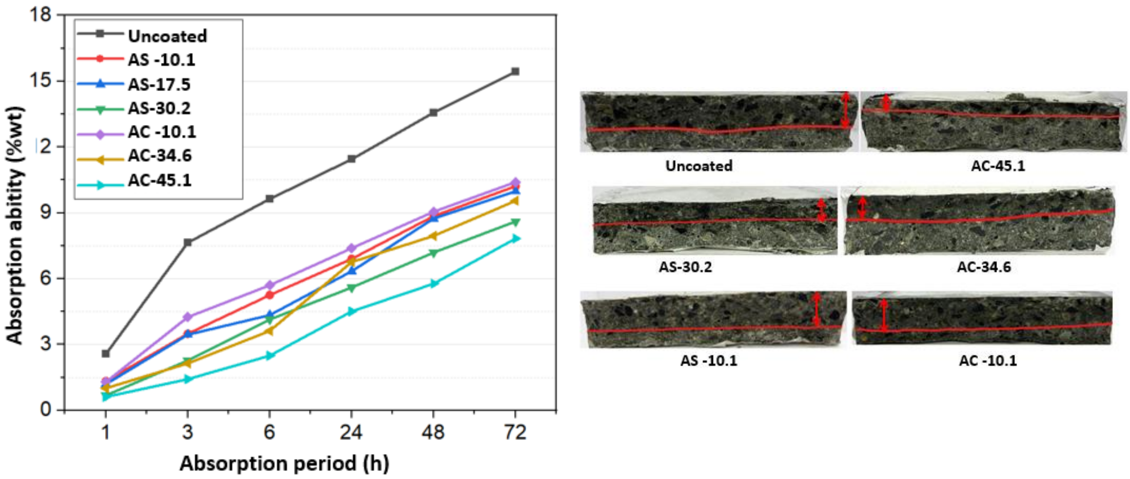


Figure 9. Water absorption of mortar sample due to capillary (note: AS = acrylic styrene; AC = acrylic).

The quantitative results presented in Figure 9 show the capillary water absorbance over 72 hours. The weight changes of the samples were monitored to measure water absorbance indirectly. The uncoated sample exhibits a high level of water absorption due to the natural surface defects, whereas the coated samples demonstrate superior impermeability. Notably, the coated sample with high solid content (AS 45.1%) exhibits the best permeability, with only 0.61% of water absorption after 3 hours and maintaining stability between 3-72 hours. This result can be attributed to the characteristics of the acrylic film, such as surface uniformity, thickness, and film strength. As emphasized in Lazar et al.'s study [4], film-forming products based on acrylic resins with high solid content exhibited superior characteristics compared to acrylic-styrene emulsions. A higher solid content leads to a lower solvent content, facilitating faster water evaporation and polymer network formation, resulting in the early formation of a more durable film.

3.4. Result of crack prevention of thin mortar slabs

Figure 10 illustrates that uncoated slabs exhibited cracks in the regulated humidity chamber's dry air environment, regardless of the mortar mixture's water-to-cement ratio. In addition, these cracks widened over time, with the average crack width being 0.4 mm for slab sample A (cement mortar with water-to-cement ratio = 0.4) and 0.65 mm for slab sample B (cement mortar with water-to-cement ratio =0.45). In the latter, several cracks were up to 1.2 mm in width. These cracks were attributed to the high water evaporation rate caused by the mortar mixture's high water-to-cement ratio, as revealed in study of Ito et al. [5] and the chamber's dry conditions, resulting in severe cracking due to plastic settlement and shrinkage**.**

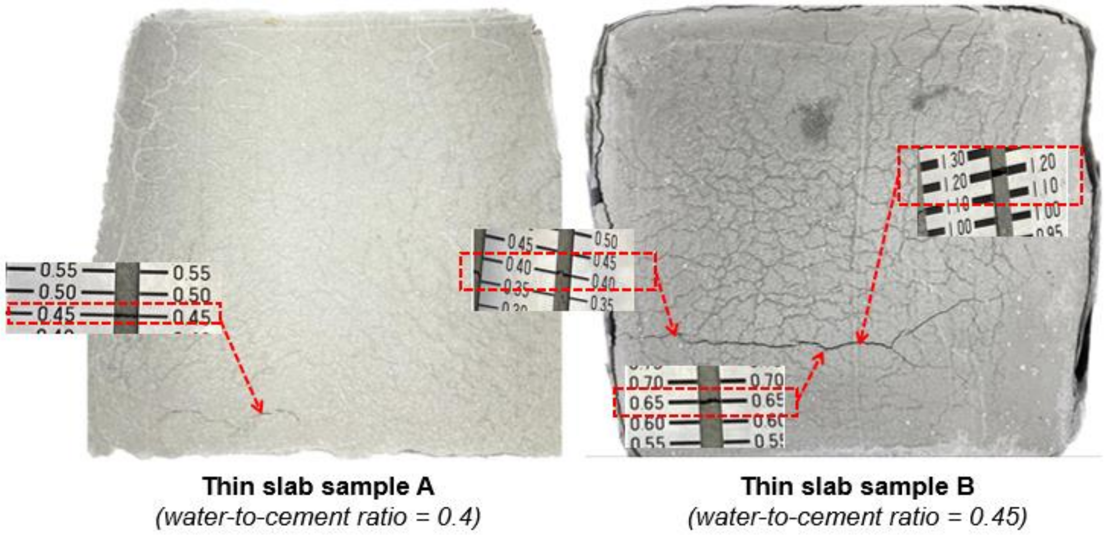


Figure 10. Two series of thin slab sample A and B with different water-to-cement ratios and generated cracks due to shrinkage during 24 hours of curing period

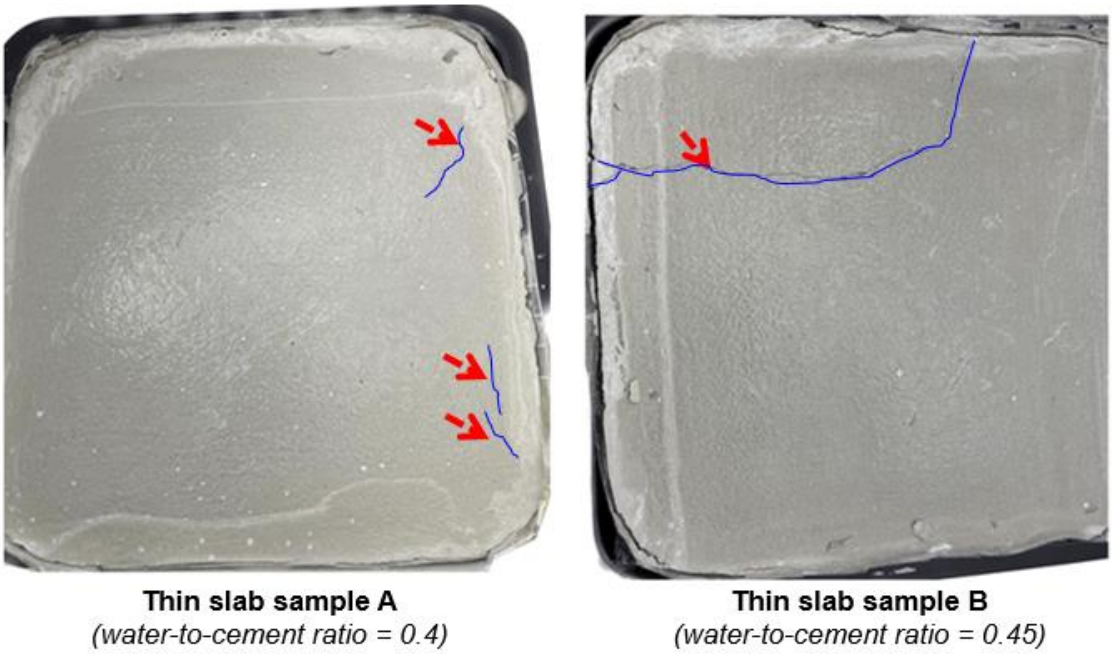


Figure 11. Two series of thin slab sample A and B with different water-to-cement ratios and generated cracks (blue) after 24 hours of curing period with sprayed water for moisturing

On the other hand, slab samples sprayed with water showed a decreasing density of cracks due to plastic shrinkage. While fewer cracks were observed for slab sample A (cement mortar with water-to-cement ratio = 0.4) and slab sample B (cement mortar with water-to-cement ratio=0.45) still showed an average crack width of 0.45 mm (Figure 11). This indicates that the water spray's moisture was insufficient to prevent the cracks generated during the curing period.

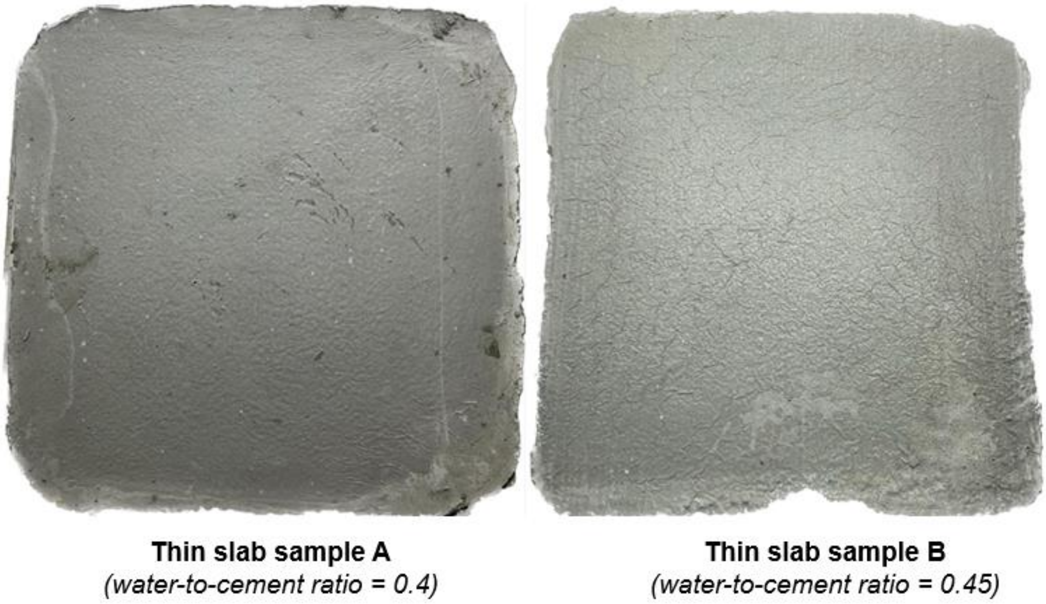


Figure 12. Two series of thin slab sample A and B with different water-to-cement ratios after 24 hours of curing period with sprayed acrylic-styrene emulsion (AS) 17,5 %wt of solid content

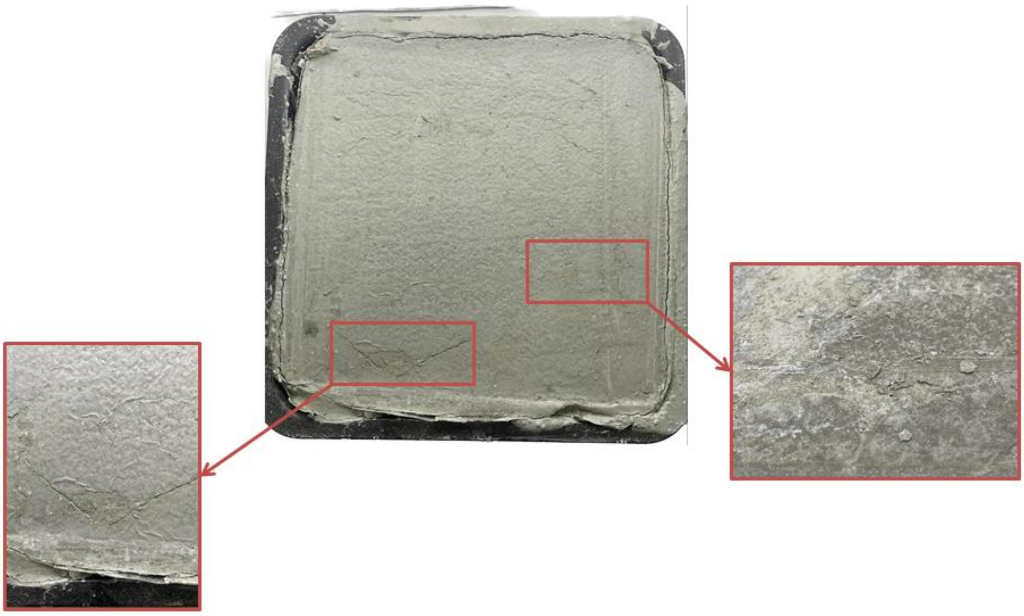


Figure 13. Visual inspection of coated sample of mortar slab with acrylic-styrene film

We observed no cracks for the coated slab samples with sprayed acrylic emulsion due to plastic shrinkage in both slab samples with water-to-cement ratios of 0.4 and 0.45 of the mortar mixture. This proves the effectiveness of the coating film in preventing free water evaporation and reducing shrinkage cracks of plasticity settlement, as also emphasized in the previsous study of Ito et al. [5]. However, we did observe an unexpected tendency for the film to fold in some positions on the slab surface (Figure 12), especially on the one cast with a higher water/cement ratio of mortar mixture (Figure 14).

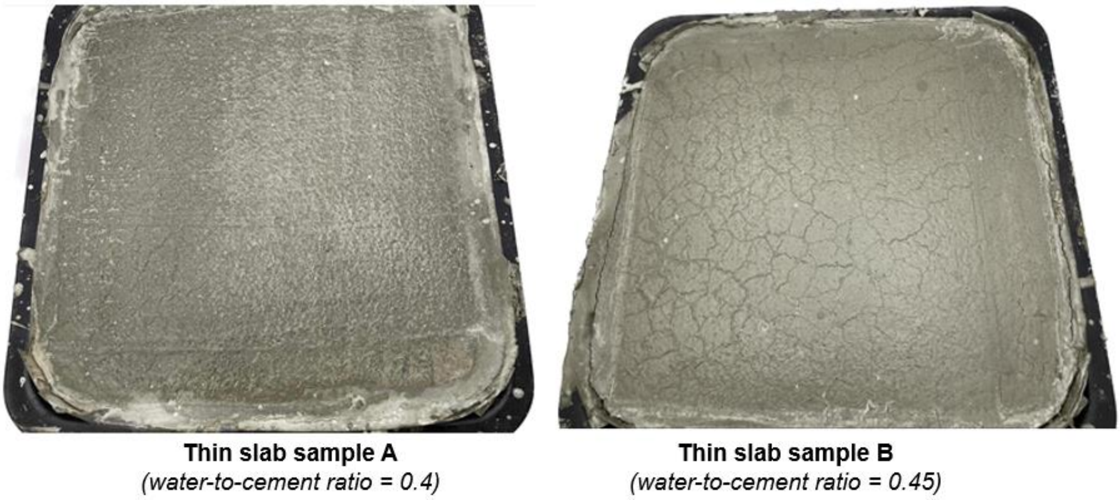


Figure 14. Two series of thin slab sample A and B with different water-to-cement ratios after 24 hours of curing period with sprayed acrylic emulsion (AC) 34.6 %wt of solid content

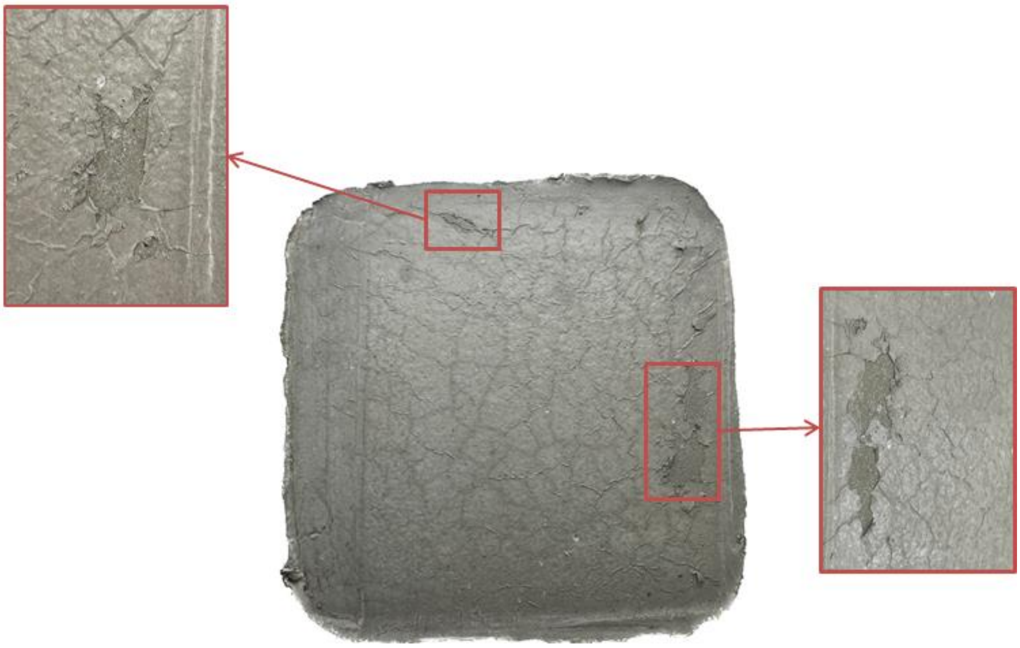


Figure 15. Visual inspection of coated sample of mortar slab with acrylic film

This folding phenomenon can be attributed to the shrinkage deformation process during the curing process, which develops the strength of the cement mortar. In samples with higher water/cement ratios, there is a greater risk of displacement of the cement mortar substrate due to plastic settlement and plastic shrinkage, which can cause folds to form due to the tensile film on the substrate surface. Meanwhile, the film is currently in the midst of evaporating water to create a coating, which puts it under strain from both procedures and ultimately results increasing.

In contrast, for slab samples A (cement mortar with lower water-to-cement ratio = 0.4), the mortar substrate dries faster, and the folding phenomenon is less pronounced because the cement mortar is relatively stable in the mold, with the free water having dried up before. The remaining water in the mortar block is essential for the cement hydration reaction, and the film plays a crucial role in retaining moisture and preventing free water evaporation. When we used a knife to slit the overlapping positions of the coating film, we did not observe any cracks on the surface of the mortar sample, which remained flat (Figure 13, Figure 15).

4. CONCLUSIONS

In conclusion, the preliminary research results on the film-forming ability and potential utilization of Rhome-Haas - Dow Chemical Vietnam’s washing waste, primarily composed of acrylic emulsion and acrylic-styrene, lead to the following conclusions:

- Material analysis found that the emulsified waste mainly contains C-O, C-H, and C=C bonds from acrylic acid and styrene, with solid contents ranging from 10 % to 45 % (wt.) for acrylic emulsion and 10 % to 30 % (wt.) for acrylic-styrene emulsion. Both emulsions can form films through water evaporation.
- The formed film effectively reduced water evaporation during the 240-minute curing period, demonstrating strong adhesion on smooth surfaces, reducing water penetration by nearly 50 % compared to uncoated mortar samples after 72 hours. The curing test for preventing cracks on thin cement mortar slabs (20mm thickness) showed significant effects after 12 hours of curing in controlled conditions (20 ± 2 °C, 43.8 ± 6.8 % relative humidity) with acrylic emulsion solution (dosage = 300g/m2). These results support the potential use of acrylic emulsion waste solution as a promising curing agent for cement concrete.

Acknowledgements. The corresponding author would like to thank Mr. Kiet from Rhomn & Haas factory - Dow Chemical Vietnam for materials support and to thank Ho Chi Minh city University of Technology (HCMUT), VNU HCM for facilities support.

CRediT authorship contribution statement. Author 1: Investigation. Author 2: Investigation, Drafting manuscript. Author 3: Investigation. Author 4: Formal analysis, Supervision. Author 5: Methodology, Supervision.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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