



Physio-chemical reactions in recycle aggregate concrete

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ABSTRACT

Concrete waste constitutes the major proportion of construction waste at about 50% of the total waste generated. An effective way to reduce concrete waste is to reuse it as recycled aggregate (RA) for the production of recycled aggregate concrete (RAC). This paper studies the physio-chemical reactions of cement paste around aggregate for normal aggregate concrete (NAC) and RAC mixed with normal mixing approach (NMA) and two-stage mixing approach (TSMA) by differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). Four kinds of physio-chemical reactions have been recorded from the concrete samples, including the dehydration of $C_3S_2H_3$, iron-substituted ettringite, dehydroxylation of CH and development of C_6S_3H at about 90 °C, 135 °C, 441 °C and 570 °C, respectively. From the DSC results, it is confirmed that the concrete samples with RA substitution have generated less amount of strength enhancement chemical products when compared to those without RA substitution. However, the results from the TSMA are found improving the RAC quality. The pre-mix procedure of the TSMA can effectively develop some strength enhancing chemical products including, $C_3S_2H_3$, ettringite, CH and C_6S_3H , which shows that RAC made from the TSMA can improve the hydration processes.

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1. Introduction

Concrete waste constitutes the major proportion of waste at about fifty percent of the total waste generated [1]. It is possible to recycle this waste by reusing them in other construction applications. An effective method would be the use of RA in the production of RAC. RA consists of stone particles attached with some old cement mortar generated in the process of crushing the demolition concrete waste while RAC is concrete, in which RA is added along with other natural ingredients.

Use of RAC fulfils three “green” requirements as set out by the World Environmental Organization: (i) it can recycle and reduce natural resources and energy consumption; (ii) it will not affect the environment; and (iii) it can maintain sustainable development. Therefore, RAC is “green” concrete. Although reuse of RA reduces the consumption of limited resources and thereby saves costs, they have some shortcomings; for examples, weak interfacial behaviour between aggregate and cement paste, and high portions of cement mortar attached that lower its quality [2–12]. To facilitate the use of RA, it is vital to identify some ways to overcome these shortcomings.

Therefore, this paper focuses on the following objectives:

- To develop a new mixing approach, TSMA, for improving RAC performance;
- To investigate the physio-chemical reactions in RAC by DSC and SEM; and
- To identify benefits possibly gained from the use of the TSMA.

2. Obstacles in the use of recycled aggregate

Although it is environmentally beneficial to use RA, the current legislation and experience are not able to support and encourage recycling demolition concrete waste [13,7]. Some technical problems, including weak interfacial transition zone between cement paste and aggregate, porosity and traverse cracks within demolition concrete, high level of sulphate and chloride contents, impurity, cement attached on RA, poor grading, and high variations in quality, render the use of RA difficult [2–5,7–12,14–27].

Since aggregate generally occupies about sixty to seventy percent of the total volume of concrete, its selection and proportioning should be given careful attention. In addition to their use as economical filler, aggregate generally provides concrete with better dimensional stability and wear resistance. In choosing aggregate for particular concrete applications, attention should be given to the three general requirements: economy of the mixture, potential strength of the hardened mass and probable durability of the concrete structure. Since RA has large open porosity, concrete pro-

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Table 1
Composition of minerals in Portland cement [40]

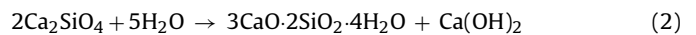
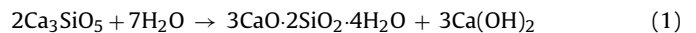
Mineral	Formula	% by weight CO ₂	SO ₃	CaO	Al ₂ O ₃
Gypsum	CaSO ₄ ·2H ₂ O	–	46.5	32.6	–
Ettringite	Ca ₆ Al ₂ (SO ₄) ₃ (OH) ₁₂ ·26H ₂ O	–	19.1	26.8	8.1
Monosulfaluminate	Ca ₄ Al ₂ (SO ₄)(OH) ₁₂ ·6H ₂ O	–	12.9	36.0	16.4
Calcite	CaCO ₃	44.0	–	56.0	–
Monocarboaluminate	Ca ₄ Al ₂ (CO ₃)(OH) ₁₂ ·5H ₂ O	7.7	–	39.5	17.9
Hemicarboaluminate	Ca ₄ Al ₂ (CO ₃) _{0.5} (OH) ₁₃ ·5.5H ₂ O	3.9	–	39.7	18.1
Hydroxyl-AFm	Ca ₄ Al ₂ (OH) ₁₂ ·7H ₂ O	–	–	40.0	18.2
Portlandite	Ca(OH) ₂	–	–	75.7	–

duced from it would be more subject to deformation and less resistant to mechanical abrasion. Density, compressive strength, modulus of elasticity, flexural strength, tensile strength, splitting tensile strength, bonding strength and shrinkage can deteriorate up to 10%, 86.4%, 50%, 16%, 6%, 50.7%, 26% and 53.4%, respectively, for RAC in comparison with NAC [28–39].

3. Cement and aggregate chemistry

At the micro-structural level, hardened cement paste (HCP) is an intimate but inhomogeneous mixture of a variety of crystalline and quasi-crystalline phases and pores of different sizes and shapes. Table 1 shows the composition of the most common minerals in Portland cement. Four major compounds: tricalcium silicate [3CaO·SiO₂ or C₃S], dicalcium silicate [2CaO·SiO₂ or C₂S], tricalcium aluminate [3CaO·Al₂O₃ or C₃A] and tetracalcium aluminoferrite [4CaO·Al₂O₃·Fe₂O₃ or C₄AF] are found in ordinary Portland cement [40–42]. In concrete, the major strength contributor is calcium silicate hydrate [CaO·SiO₂·H₂O, abbreviated as CSH], the principal hydration product, which is a highly disordered crystalline material of variable composition. Upon the addition of water, C₃S rapidly reacts to release calcium ions, hydroxide ions, and a large amount of heat. The pH value rises quickly to greater than 12 because of the release of alkaline hydroxide (OH⁻) ions. This initial hydrolysis slows down quickly after it starts, resulting in a decrease in heat evolved. The reaction continues slowly and produces calcium and hydroxide ions, until the system becomes saturated. Once this occurs, the calcium hydroxide [Ca(OH)₂, abbreviated as CH] starts to crystallize. Simultaneously, CSH begins to form. Ions precipitate out of solution, which accelerates the dispersion of C₃S to calcium and hydroxide ions. The evolution of heat is then dramatically increased. The formations of CH and CSH crystals provide ‘seeds’ upon which more CSH can form. The CSH crystals grow thicker making it more difficult for water molecules to reach the unhy-

drated C₃S. The speed of the reaction is now controlled by the rate at which water molecules diffuse through the CSH coating. This coating thickens over time, causing the production of CSH to become slower and slower. The molecular structure of CSH is layered and provides for a very high internal porosity. The process of the hydration of C₃S is given in Eq. (1) [43]. C₂S also affects the strength of concrete through its hydration. C₂S reacts with water in a similar manner of C₃S, but much more slowly. The heat released is less than that generated by the hydration of C₃S because the C₂S is much less reactive. The process of the hydration of C₂S is given in Eq. (2) [43]:



In contrast to CSH, Ca(OH)₂ [CH], also known as Portlandite, is highly crystalline and has a fixed composition, which comprises more than 20% of the hydration products in a fully cured cement paste [44]. CH appears as thin hexagonal platelets, often layered, typically tens of μm across. With continued hydration, they grow massively, lose their hexagonal outline, and encapsulate other regions of the paste [29]. Massive blocks of CH crystals can be easily identified in mature pastes. Unhydrated residues of clinker grains, which are crystalline in nature, are present even in well-hydrated systems. They are produced by the hydration of the silicate phases, C₃S, C₂S, and free lime [CaO] as shown in Eqs. (3)–(5) [44,45]. It can be seen from Eqs. (3) and (4) that the weight for the hydration of C₃S (Eq. (3)) is three times the amount of CH as does the hydration of C₂S (Eq. (4)). Thus the ratio of these two phases will affect the amount of CH formed.

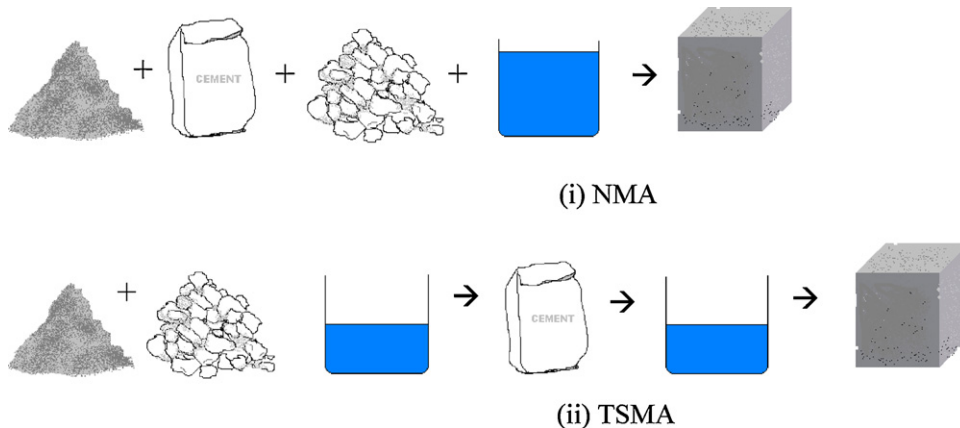
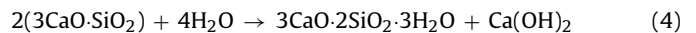
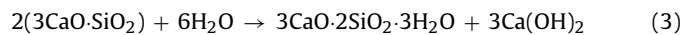


Fig. 1. Mixing procedures of (i) normal mixing approach and (ii) two-stage mixing approach.

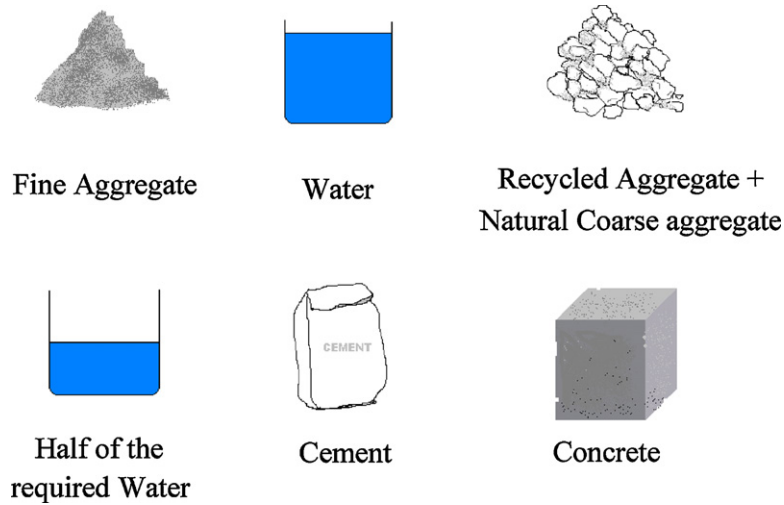
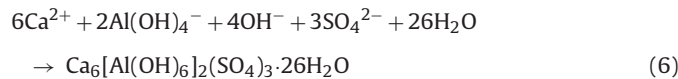


Fig. 2. Symbols used for representing various materials.

Table 2
Mix proportions

Ingredients of concrete	Mass (kg/m ³)
Ordinary Portland cement	100
Fine aggregate	180
20 mm coarse aggregate	180
10 mm coarse aggregate	90
Water	45



Another type of crystal commonly found in HCP is ettringite [Ca₆[Al(OH)₆]₂(SO₄)₃·26H₂O], as shown in Eq. (6). Where ettringite is observed in cracks and voids, it appears as needles that have a distinctive low birefringence and low refractive index; it is often found in conjunction with CH [44,45]. Situations such as movement of water through concrete or the formation of alkali-silica gel appear capable of causing conditions that allow it to reform. In old concrete, these deposits can be massive. However, as the ettringite is reformed in void spaces, its expansion can be accommodated [44,45]. Ettringite is produced by a reaction that requires an excess of the sulphate ion SO₄²⁻ over the aluminate phase in the pore solution [44,45]. As hardening proceeds, more of the aluminate phase moves into the pore solution, which converts the ettrin-

gite to mono-calcium sulfo-aluminate [CaSO₄·2CaO·Al₂O₃·12H₂O]. This conversion results in a small decrease in volume and thus may occur in the hardened paste without damage. Both ettringite and monocalcium sulfo-aluminate hydrate formed during setting and hardening are usually submicroscopic and require the use of scanning electron microscopy (SEM) observations. Only where ettringite is secondary and reformed from reactions of the SO₄²⁻ ion with monocalcium sulfo-aluminate do its crystals become large enough to be observed in thin-section. Localized concentrations of ettringite are controlled by both the amount of water and space at particle surfaces, with crystallization modified by the surface texture and porosity of the aggregate, which greatly improves the strength [44,45].

4. Materials and methods

To improve RAC quality, a new mixing method: TSMA is developed by Tam et al. [46], which divides the mixing process into two parts and proportionally splits the required water into two which are added at different timing. Fig. 1 illustrates the NMA and TSMA mixing procedure while Fig. 2 shows the symbols used.

RA used for the laboratory experiments are collected from the Hong Kong government centralized recycling plant with average absorption values of 1.65% for 20 mm and 2.63% for 10 mm aggregate. The RA quality is slightly poorer than that of virgin aggregate which has water absorption values of about 0.77% and 0.57% for 20 mm and 10 mm aggregate, respectively. For the chemical com-

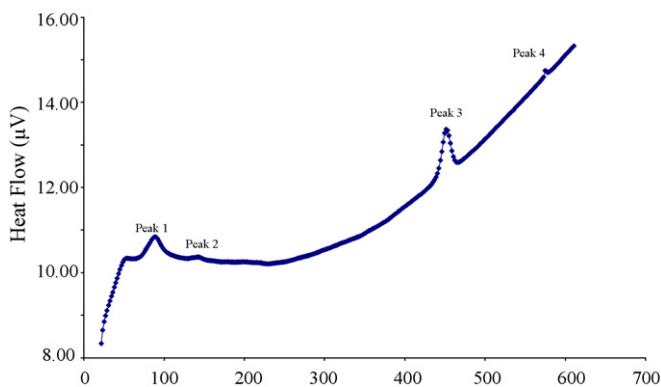


Fig. 3. DSC curve for the NAC with 0% RA substitution in the NMA.

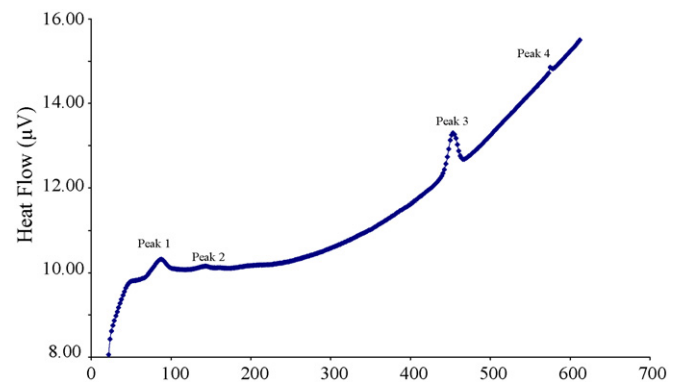


Fig. 4. DSC curve for RAC with 100% RA substitution in the NMA.

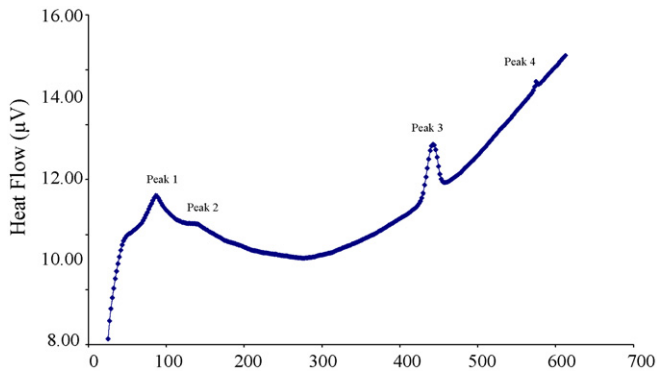


Fig. 5. DSC curve for NAC with 0% RA substitution in the TSMA.

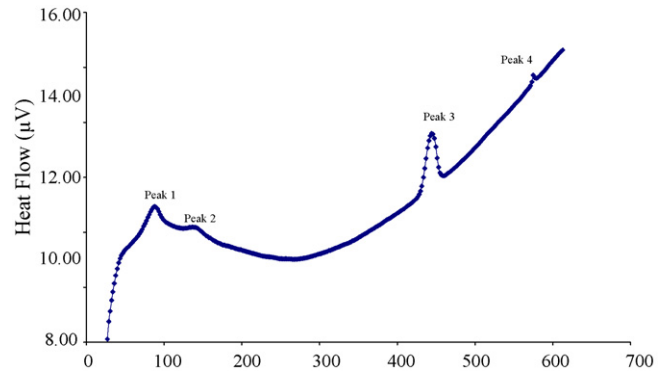


Fig. 6. DSC curve for RAC with 100% RA substitution in the TSMA.

positions of the RA, the chloride content is about 0.0021% for 10 mm and about 0.0070% for 20 mm and the sulphate content is about 0.008%, compared with natural aggregate of only 0.0012% for 10 mm and 0.0016% for 20 mm for chloride content and of 0.003% for sulphate content.

The designated mix proportions follow that stipulated in the specifications of the Buildings Department (BD) of the Hong Kong Special Administrative Region [47] with a water to cement ratio of 0.45 (see Table 2).

In this paper, four types of concrete samples are produced for investigation: (i) virgin aggregate with the NMA; (ii) 100% RA substitution with the NMA; (iii) virgin aggregate with the TSMA; and (iv) 100% RA substitution with the TSMA.

Concrete samples made from the above four experiments are investigated using DSC [Seiko Instruments (SII) SSC 5200 TG/DTA 220]. Before collection of samples from the cube, the first 4–5 mm of the cube surface is grinded off to avoid the ambient humidity influencing the moisture content of samples. These samples are placing in a small and flat container in shallow pans, with the aim of making a good thermal contact among sample, pan and heat flux plate. Cement pastes around aggregate from the four experiments are collected for investigation. The samples used weighted about 40 mg (the maximum allowed weight) are collected from cubes stored under water at about 20 °C. The samples are then heated up to about 620 °C in a constant heating rate of about 20 °C/min, in a dynamic helium atmosphere to develop DSC curves for all samples.

The microstructure of the samples is investigated using scanning electron microscopy. The samples are cut into 2 mm thick plates; the fracture surface is gold-plated for SEM examination by a JEOL JSM-820 scanning microscope.

5. Results and discussions

Physio-chemical reactions of the cement paste around aggregate from NAC and RAC mixed with the NMA and TSMA are investigated. In principles, four compounds should exist in normal cement paste [48,49]; they are: (i) tricalcium silicate (C_3S); (ii) dicalcium silicate (C_2S); (iii) tricalcium aluminate (C_3A); and (iv) tetracalcium aluminoferrite (C_4AF). At the early stage of chemical reactions, C_3S and C_2S react with water (H_2O) to produce calcium silicate hydrate ($C_3S_2H_3$) and calcium hydroxide (CH) [50]. Eqs. (7) and (8) show the reactions:



When the temperature is lower than 620 °C, there are four physio-chemical reactions in the concrete: (i) dehydration of $C_3S_2H_3$; (ii) dehydration of iron-substituted ettringite; (iii) dehydroxylation of CH; and (iv) solid-state transformation of C_6S_3H , which form four absorption peaks in DSC curves at about 90 °C, 135 °C, 441 °C and 570 °C, respectively, as shown in Figs. 3–6 for the samples. Table 3 shows the summary of the DSC results.

From the DSC results, NAC has higher summation values of heat flow (Δ) for Peaks 1 and 3 than that in RAC for either NMA or TSMA (see Table 3). For example, about 0.13 and 0.12 are for 0% RA substitution and 100% RA substitution respectively for the NMA for Peak 1. This indicates that the old cement paste attached to the RA may have absorbed more water from the mix and the new cement paste is reacted with the old ones, both lowering the strength of RAC produced. With the same water to cement ratio, mixing approach and curing condition, RAC will require higher amount of water, which

Table 3
Peak areas from the DSC curves

Figure	Element	Peak area			
		Peak 1	Peak 2	Peak 3	Peak 4
0% RA in NMA	max. (μV)	+0.59	+0.49	+1.08	+1.37
	Δ (μV)	+0.13	+0.05	+0.21	+0.055
	Temperature (°C)	90	135	441	570
100% RA in NMA	max. (μV)	+0.47	+0.43	+1.08	+1.38
	Δ (μV)	+0.12	+0.06	+0.20	+0.055
	Temperature (°C)	90	135	441	570
0% RA in TSMA	max. (μV)	+0.56	+0.46	+0.74	+0.97
	Δ (μV) ^a	+0.15	+0.07	+0.23	+0.06
	Temperature (°C)	90	135	441	570
100% RA in TSMA	max. (μV)	+0.51	+0.41	+0.77	+0.99
	Δ (μV)	+0.13	+0.07	+0.22	+0.06
	Temperature (°C)	90	135	441	570

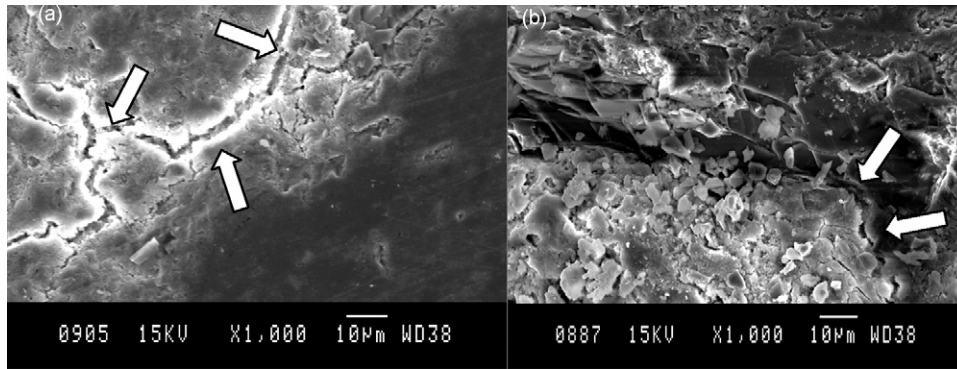


Fig. 7. Cracks in cement paste remains of RA.

thus lower the quality and strength of concrete when compared to those of NAC. From the SEM graphs (see Fig. 7), it is found that the RA exhibits more pores and cracks, making it having a high water absorption value [50,46]. However, during setting and hardening, the new cement paste will first react with the old cement paste attached to the RA, in which it will use some of the required water. It is the main reason that affects and reduces the formation of $C_3S_2H_3$, ettringite, CH and C_6S_3H , leading to poorer strength of RAC.

From the DSC curves for the TSMA, it shows that there is an endothermic curve (a downward budging curve) occurred after Peak 2, which reaches its minimum at about 260°C while this phenomenon does not exist in the NMA. Together with the results of SEM, this phenomenon may be resulted from the formation of hillebrandite (C_2SH), which is formed at around $140\text{--}350^\circ\text{C}$. The SEM analysis shows the different development processes of CH crystals; for the TSMA, the CH crystals are rougher and larger in size resulted from the existence of large amount of micro-cracks on the surface of RA (see Fig. 8).

In comparing the results of NMA and TSMA, the summation values of heat flow (Δ) for the TSMA are higher than those for the NMA for all peaks. For example, 0.13, 0.05, 0.21 and 0.055 for Peaks 1–4, respectively, have been recorded for 0% RA substitution in the NMA while 0.15, 0.07, 0.23 and 0.06 for Peaks 1–4, respectively, have been recorded for 0% RA substitution in the TSMA (see Table 3). It can be inferred that the activity of the cement paste on the surface of RA is high (see Fig. 8). The pre-mix procedure of the TSMA can effectively develop some strength enhancing chemical products:

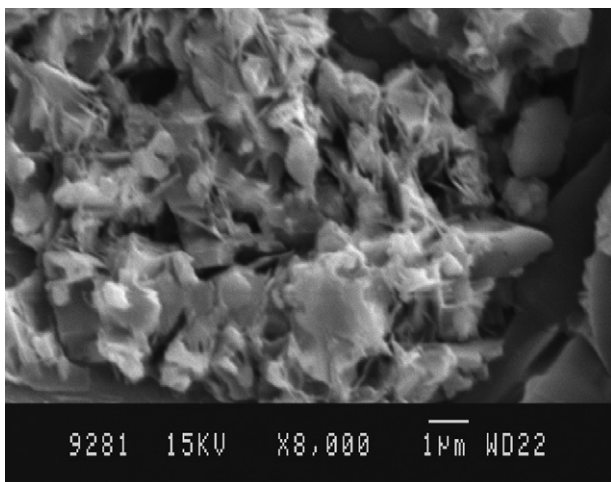


Fig. 8. Development of $C_3S_2H_3$ after mixing by the TSMA under the SEM examination.

$C_3S_2H_3$, ettringite, CH and C_6S_3H . The cement paste at the interfacial zone for RAC is also chemically active under the NMA; however, the hydration products developed cannot contribute much to the strength development; these products are in fact puffy in nature and thus even leading to reduction in strength. This shows that RAC made from the TSMA can reduce the effect of this phenomenon and improves the hydration process.

6. Conclusion

This paper has studied the physio-chemical reactions of cement paste around aggregate for normal aggregate concrete (NAC) and recycled aggregate concrete (RAC) mixed with the normal mixing approach (NMA) and the two-stage mixing approach (TSMA) by differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). Four kinds of physio-chemical reactions have been recorded in the concrete samples, including the dehydration of $C_3S_2H_3$, iron-substituted ettringite, dehydroxylation of CH and development of C_6S_3H at about 90°C , 135°C , 441°C and 570°C , respectively. From the DSC results, it was found that the concrete samples with recycled aggregate substitution have lower values of all the four physio-chemical reactions than those without recycled aggregate substitution. The major reason of the activeness of the physio-chemical reactions occurred in the RAC is the large amount of mini-cracks in the recycled aggregate. The mini-cracks absorb some of the required water and reduce water amount for mixing; thus hinder the development of $C_3S_2H_3$, ettringite, CH and C_6S_3H and reduce the concrete strength. Therefore, the strength of the surface cement paste in RAC is lower than that in NAC; and thus it reduces the RAC quality. Despite of the poorer quality found for RAC, the use of TSMA can improve the RAC quality. From the DSC results, it is found that the TSMA has a higher summation values for all the physio-chemical reactions than that in the NMA with the same RA percentage substitution. The pre-mix procedure of the TSMA can effectively develop some strength enhancing chemical products, $C_3S_2H_3$, ettringite, CH and C_6S_3H . The cement paste at the interfacial zone for RAC is also chemically active under the NMA. However, the hydration products developed cannot contribute much to the strength development and these products are in fact puffy in nature and thus even leading to reduction in strength. This shows that RAC made from the TSMA can reduce the effect of this phenomenon and improves the hydration processes.

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