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Research Article

Sodium Silicate Gel Effect on Cemented Tailing Backfill That Contains Lead-Zinc Smelting Slag at Early Ages

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This paper presents the results of an experimental study on the priming effect of sodium silicate gel (SS) on cemented tailing backfill (CTB) that contains lead-zinc smelting slag. CTB and cemented paste (CP) containing lead-zinc smelting slag samples with SS of 0 and 0.4% of the mass of the slag were prepared and cured at 20°C for 1, 3, 7, and 28 days. Mechanical test and pore structure analyses were performed on the studied CTB samples, microstructural analyses (X-ray diffraction analysis and thermal gravity analysis) were performed on the studied CP samples, whereas the electrical conductivity of CTB was monitored. The results reveal that SS has a significant positive effect on cementitious activity of binder mixed by cement and lead-zinc smelting slag. This activation leads to the acceleration of binder hydration process, the formation of more cement hydration products in the CTBs, and the refinement of their pore structure, which is favorable for the strength development of CTB.

1. Introduction

It is known that cemented tailing backfill (CTB) technology is a common method for ground support and mine waste disposal during mining operations [1–5]. CTB is a mixture of dewatered tailings, a hydraulic binder, and water [2, 3]. Usually, the dewatered tailing is accounted for a high proportion between 70% and 85%, and the hydraulic binder only occupies a small proportion between 3 and 7 wt.%. Ordinary Portland cement type I (PCI) is usually used as a binder in CTBs, and the binder consumption can account for up to 75% the cost of CTB [5]. Many previous studies [6, 7] try to reduce the cost as well as improve the properties (such as microstructure and strength) of CTB by a partial replacement of Portland cement by ground-granulated blast furnace slag (GGBFS), nonferrous smelting slag, or fly ash. Lead-zinc smelting slag is a low-cost by-product which can be a pozzolanic material and reacts with water and lime to produce cementitious products [8–10].

Sodium silicate gel (SS), $Na2SiO_3{\cdot}9H_2O$, is a kind of water-soluble silicate which appears as crystallization of orthogonal double cone or white-to-gray lump or powder. SS is often used as a binder and an activator in cement-based materials to improve the strength and stability [11]. For example, after contacted with water, a waterproof layer of aluminum silicate film, which can prevent further hydration reaction of GGBFS, can be created on slag particles. As a kind of alkali excitation agent, SS can speed up the disintegration of GGBFS vitreous and promote the hydration process [12]. However, the effect of SS on the binder containing lead-zinc smelting slag has not been explored.

Early age (≤28 days) mechanical strength, which controls the mechanical stability and supporting ability of CTB structure, is considered as one of the most important performances of CTB. To ensure safe underground working conditions, different mechanical strengths are required depending on the function and application of the backfill. For example, up to 1 MPa is needed for free-standing wall, and strength values higher than 4 MPa are required for roof support [13–15]. UCS test is commonly performed to test the mechanical strength and evaluate the stability of CTB [2, 3]. As well known, high early strength of CTB can provide sufficient early mechanical stability to CTB structures which can reduce the mining cycle and increase the mine productivity as well as significantly reduce or eradicate the risk of liquefaction [6].

Considering the current situation mentioned above, the main purpose of this paper is to experimentally research and present the priming effect and the effect of sodium silicate gel (SS) on CTBs that contain lead-zinc smelting slag at early ages.

2. Experimental Programs

2.1. Materials

2.1.1. Binder. Portland cement type I (PCI) blended with grounded lead-zinc smelting slag in the weight ratio of 50/50 was used as a binder in all samples. Grain size distribution analyses were conducted by laser particle size analyzer, a Mastersizer 2000. Analytical analyses were conducted on the nonferrous slag by using X-ray fluorescence spectroscopy. The apparatus is Philips Pw2400. Determination of the specific gravity of the tailing particles is in accordance with ASTM D854. Figure 1 shows the particle size distribution of grounded lead-zinc smelting slag and commercial ground blast furnace slag on Canadian market. The main chemical properties and relative density of the studied lead-zinc smelting slag are presented in Table 1. Table 2 shows the chemical composition of the Portland cement and relative density.

2.1.2. Tailings. Figure 1 shows the particle size distribution of the tailings used in this study. The main chemical properties and relative density of the tailings are presented in Table 3.

2.1.3. Sodium Silicate Gel. Powdered sodium silicate gel was used in order to be completely mixed with tailing and binder particles.

2.1.4. Mixing Water. Distilled water (DW) was used in this study.

2.2. Sample Preparation and Mix Proportioning. All CTB samples were prepared with a water-to-binder ratio of 7 and a binder content of 4.5% in terms of mass. Weighted tailings, binder, SS of 0 and 0.4% of the mass of the lead-zinc smelting slag, and mixing water were mixed and homogenized until obtaining a homogeneous paste. The produced mixes were poured into concrete curing cylinders, 5 cm in diameter and 10 cm in height. The specimens were then sealed (this avoids the evaporation of water) and cured at 20°C for periods of 1, 3, 7, and 28 days. Cylinders sized 10 cm in diameter and 20 cm in height were used for monitoring tests. Samples of lead-zinc smelting slag cement paste (SCP), with a water-cement (w/c) ratio of 2, were prepared in the same procedure for microstructural analysis.

2.3. Testing of Specimens

2.3.1. Mechanical Tests. Following ASTMC 39, UCS tests were performed on the CTB specimens. The loading capacity and load rate are 50 kN and 1 mm/min, respectively. Each test was repeated at least twice, and the average was chosen as the strength of the tested sample.

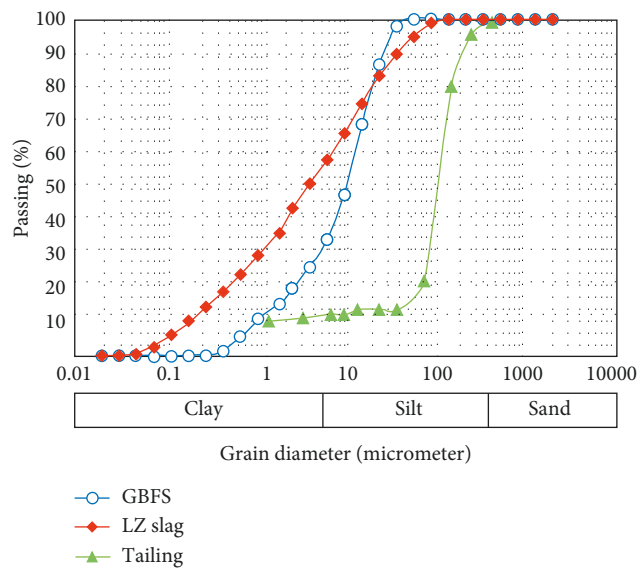


Figure 1: Grain size distribution curve of the tailing material used.

2.3.2. Microstructural Analysis. To assess the SS effect on binder hydration products in the CTBs, a series of microstructural analyses that included examination of the pore structure and evolution of binder hydration products were carried out to evaluate the microstructural properties of the studied CTB. The microstructure of the studied CTB samples was investigated by mercury intrusion porosimetry (MIP) and thermal (TG/DTG) and XRD analyses. MIP tests were performed by PMI mercury/nonmercury intrusion porosimeter to evaluate the pore size distribution and the total porosity. The CTB samples prepared for the MIP tests were cured up to 28 days. Prior to tests, all samples were first oven-dried at 50°C until mass stabilization. Drying at this temperature did not appear to cause cracking.

2.3.3. Electrical Conductivity Monitoring. Electrical conductivity (EC) monitoring, which can assess the progress of cement hydration that occurs within the hydrating cementitious materials, was performed on the CTB samples. An EC sensor (5TE) was placed in the middle of the CTB samples to measure the EC evolution. All the sensors are connected to a data logger (Em 50) so that the data could be recorded.

3. Results and Discussions

3.1. Strength Evolution of CTBs at Early Ages. Active effect of sodium silicate gel on cemented tailing backfill that contain lead-zinc smelting slag can be seen in the strength evolution of CTBs at early ages. Figure 2 shows the UCS evolution with curing time of the prepared CTBs with SS and without SS, respectively. It can be observed that the CTB, whose lead-zinc smelting slag is activated by SS, shows significantly higher 28 days UCS than CTB that does contain SS.

Table 1: Main chemical compositions of the studied lead-zinc smelting slag.

Element unit	SiO_2 (wt.%)	Fe_2O_3 (wt.%)	Al_2O_3 (wt.%)	TiO_2 (wt.%)	CaO (wt.%)	MgO (wt.%)	Na_2O (wt.%)	K_2O (wt.%)	MnO (wt.%)	P_2O_5 (wt.%)	LOI (wt.%)
	15.5	30.41	6.20	0.284	2.92	0.55	0.35	0.50	0.47	0.15	40.24

Table 2: Main chemical compositions of the cement used.

Element unit	SO_3 (wt.%)	Fe_2O_3 (wt.%)	Al_2O_3 (wt.%)	SiO_2 (wt.%)	CaO (wt.%)	MgO (wt.%)	Relative density
PCI	3.82	2.70	4.53	18.03	62.82	2.65	3.1

Table 3: Main mineral compositions of the tailings used.

Mineral (wt.%)	Quartz	Calcite	Magnetite	Muscovite	Albite
Content (%)	0.52	0.16	0.05	0.35	99.00

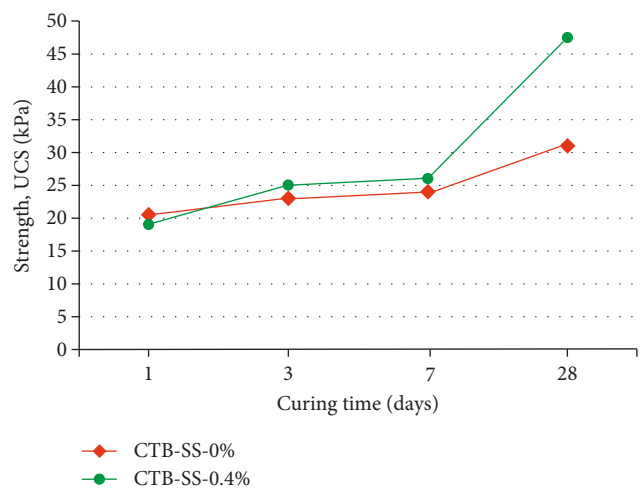


Figure 2: Strength evolution of CTB for different SS contents at early ages.

This higher UCS value can be explained as the result of the fact that the sodium silicate acts as an alkaline activator of the lead-zinc smelting slag, which in turn results in the stimulation of the hydration of lead-zinc smelting slag and thus contributes to the strength development of CTB. In summary, the results presented above suggest that the activation of the lead-zinc smelting slag by SS can significantly increase the UCS of CTB. This activation effect of the SS is due to the combined effects of the following factors: (i) the refinement of the pore structure of CTB; (ii) the formation of more hydration product (in particular, calcium hydroxide); and (iii) the acceleration of binder hydration. These factors can be supported by the research results as discussed below.

3.2. Refinement of the Pore Structure. Sodium silicate gel caused a refinement influence on the pore structure which significantly affects the strength of CTB. Generally, finer pore structure is associated with higher strength of the porous medium [16] or cemented backfill [4, 17]. Figure 3 shows the MIP results performed on CTBs with 0% and 0.4% SS and

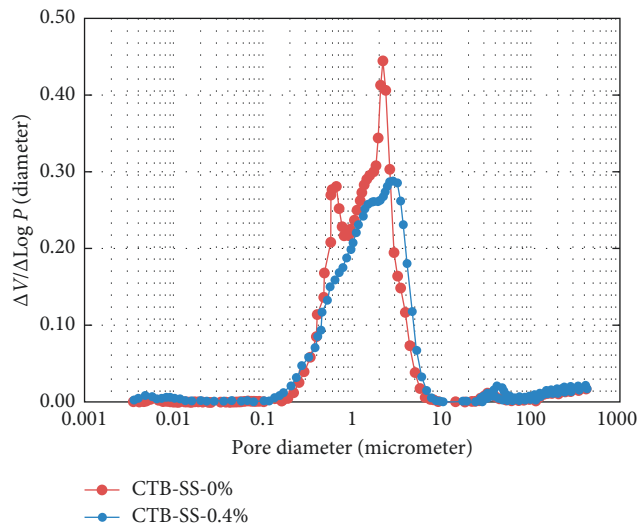


Figure 3: Differential pore size distribution curves of CTB that contain lead-zinc slag with SS and without SS.

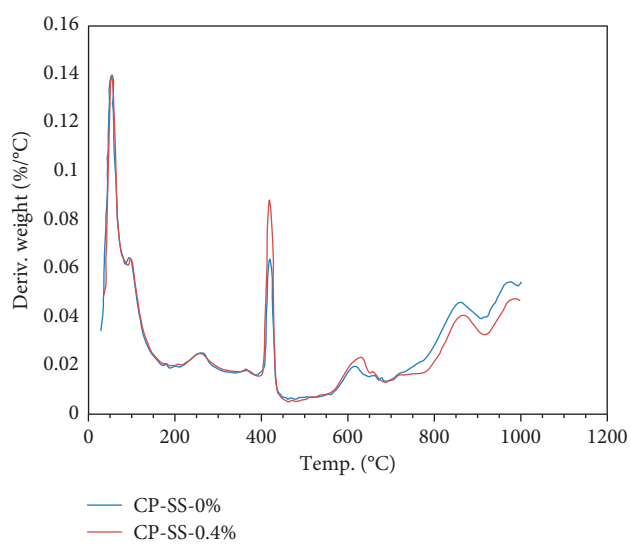
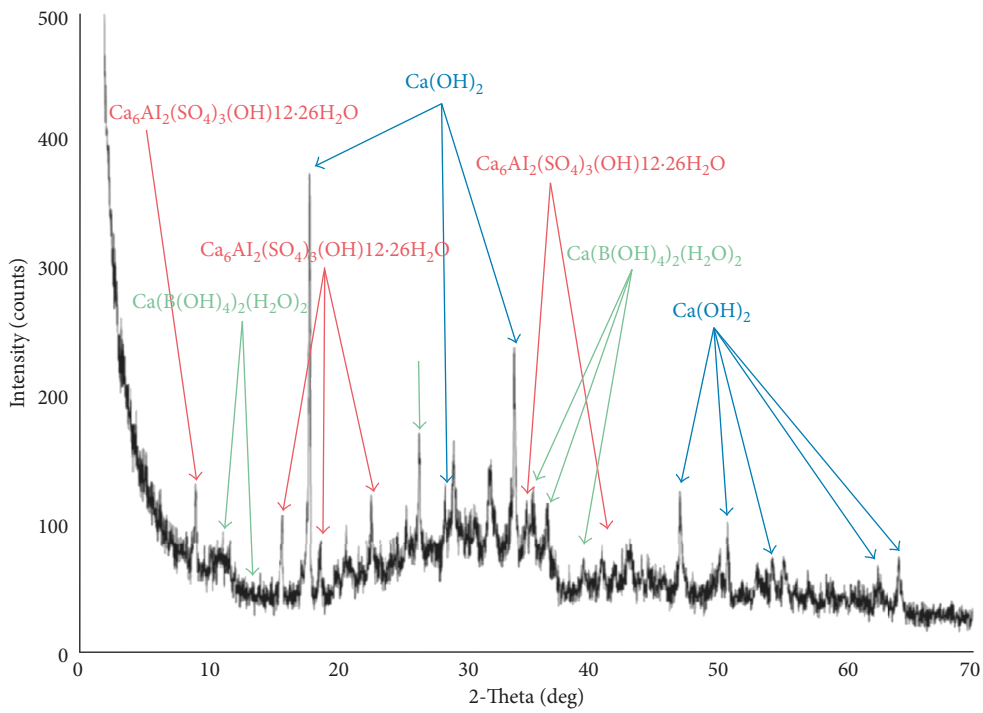
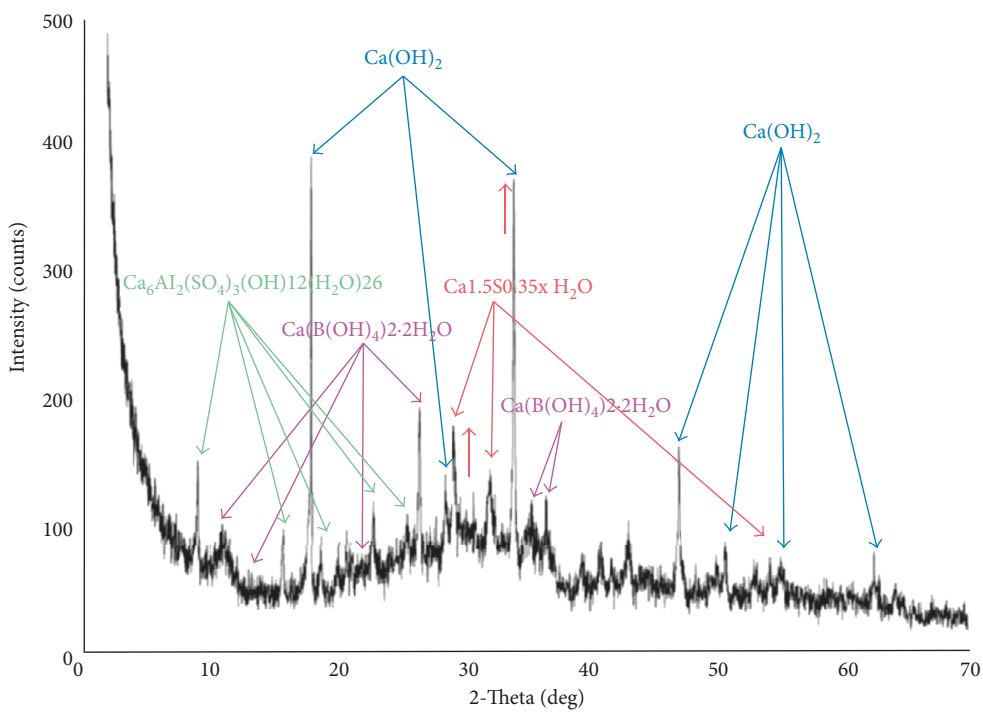


Figure 4: TG (a)/DTG (b) diagrams for FA-cemented paste of FA-CTB cured for 28 days.

cured for 28 days. From this figure, it can be observed that the SS has a significant influence on the pore size distribution of CTBs containing lead-zinc smelting slag. It is clear that the CTB that contains SS has a finer pore structure than that



(a)



(b)

Figure 5: XRD results of (a) CTB without SS and (b) CTB that contains SS.

without the SS. This finer pore structure is attributed to the precipitation of higher amount of binder hydration products in the CTB that contains SS due to the activation of the binder by SS. This higher amount of binder hydration product is also supported by the results of TG/DTG and XRD analyses presented below.

3.3. Formation of More Hydration Products. More amounts of hydration products were formed due to the addition of SS in CTBs. The results of the thermal analyses are presented in Figure 4. This figure presented the TG/DTG diagrams of cement pastes of CTB that contain lead-zinc smelting slag with SS (CP-SS-0.4%) and without SS (CP-SS-0%).

The DTG localizes the ranges that correspond to the thermal decompositions of the different phases in the paste, while TG simultaneously provides the weight loss due to these decompositions. This figure shows, globally, three main peaks associated with rapid weight loss and major phase transformations. It is well known that the weight loss or peak situated in the 50–200°C temperature range results from the dehydration of C-S-H, ettringite, carboaluminates, gypsum, etc. [18], whereas the weight loss or a peak located at 400–450°C and 600–750°C is mainly caused by the decomposition of CH and calcite, respectively [19, 20]. The comparison of the TG/DTG diagram of the cement pastes activated with SS and that of the non-SS shows that the peaks or weight losses are higher for cement paste activated with SS. These experimental observations indicate that larger amounts of hydration products are formed in the binder activated with SS than in the nonactivated binder. This larger amount of binder hydration products contribute to give the CTB that contains lead-zinc smelting slag higher strength. These results confirm the ability of SS to activate the studied binder that contains lead-zinc smelting slag.

The formation of more hydration products related to SS can be demonstrated by the XRD results of SCP samples that contain lead-zinc smelting slag activated by SS and without SS and cured at 28 days, which are presented in Figures 5(a) and 5(b). These results show clearly that higher amounts of binder hydration products are produced in the CTB sample that contains lead-zinc smelting slag activated with SS than in the CTB without SS. Indeed, a comparative analysis of the XRD results presented in Figures 5(a) and 5(b) indicates that the peaks of CH, especially at 18 and 34 degrees 2-theta, are higher in the binder activated by SS than in the binder without SS. This confirms again the ability of the SS to activate the CTBs that contain lead-zinc smelting slag.

3.4. Acceleration of Binder Hydration Process. Finally, SS shows obvious acceleration of binder hydration in CTBs that contain lead-zinc smelting slag. Figure 6 presents the electrical conductivity evolution of CTBs with SS and without SS. Variations in the electrical conductivity (EC) can be credited to changes in the number and/or the mobility of charge carrying ions and thus can be used as an indicator for cement hydration characteristics as well as for tracking the structural changes that occur in hydrating cementitious materials. The value of EC of cementitious materials is affected by the ion concentration in the pore system. Furthermore, it is recognized that the cement hydration process results in the formation of C-S-H, CH (portlandite), ettringite, and other compounds. During hydration, the capillary pores in hardening cement paste are progressively filled up with hydration products and the solid phases form a rigid microstructure with increasing strength. Then, electrical conductivity of cement paste reduces with time. From this figure, it can be observed that the electrical conductivity peak shift to shorter time as the addition of SS, which indicates an acceleration effect of the binder hydration. Actually, the electrical conductivity of CTB without SS reaches the peak value of 3.24 mS/cm after about 15 hours, while the CTB with SS reaches the peak value of 4.41 mS/cm only about 5 hours after mixing. It can be concluded that SS not only accelerated the hydration process but also enhanced the reaction rate in CTBs that contain lead-zinc smelting slag.

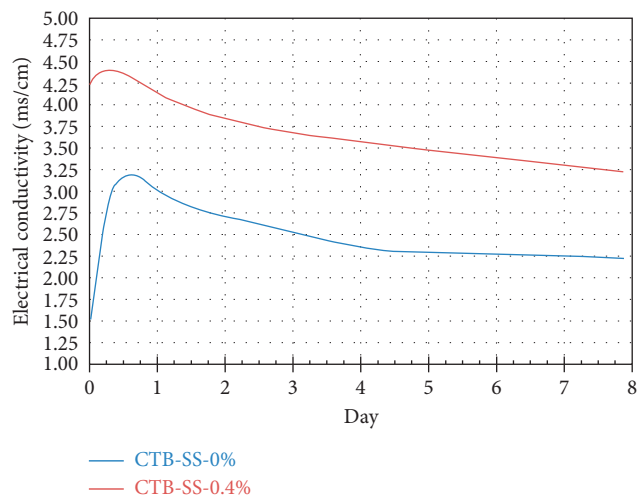


Figure 6: Results of electrical conductivity monitoring of CTB that contains NFS with SS and without SS.

4. Summary and Conclusions

This paper presents the experimental results of a study which focuses on the effect of SS on CTBs that contain lead-zinc smelting slag. It is found that SS can cause an activation effect and increase the strength value of CTB made of Portland cement and lead-zinc smelting slag. This activation includes the refinement of the pore structure of CTB, the formation of more cement hydration products in the CTBs, and the acceleration of the binder hydration process, which has been confirmed by the results of microstructural analyses (MIP, TG/DTG, and XRD) and electrical conductivity (EC) monitoring, respectively. It can be concluded that the SS can cause an active effect on CTBs that contain lead-zinc smelting slag.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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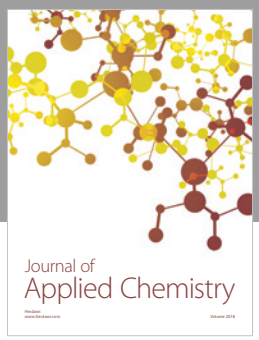

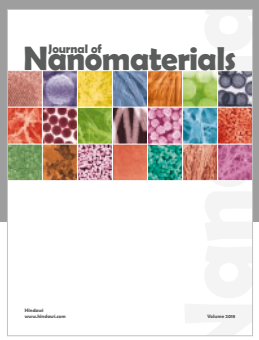

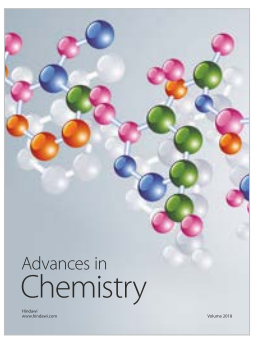

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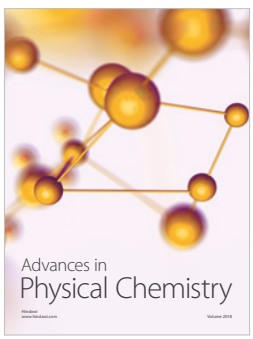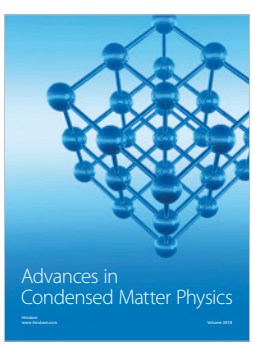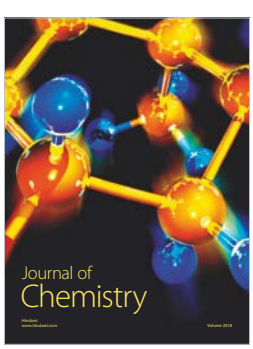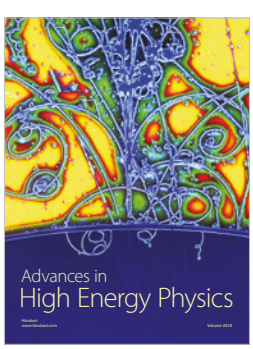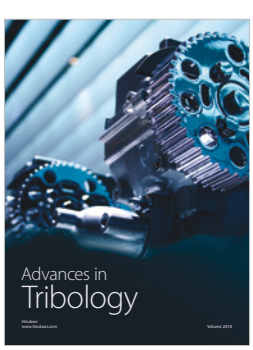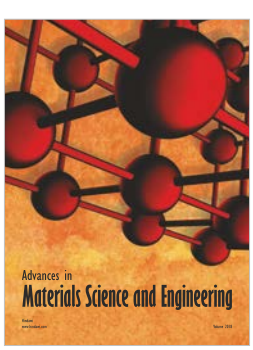
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