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Title: **SYNTHESIS AND RESEARCH OF NEW MODIFIED POLYMER SEROBETONE**

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SYNTHESIS AND RESEARCH OF NEW MODIFIED POLYMER SEROBETONE

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Abstract: Sulfur is the third most abundant chemical element in oil at a concentration of more than 10 wt%, and its extraction from oil and gas processing is carried out in accordance with environmental restrictions.

Keywords: fuel, sulfur, calcium oxide, chemical modifiers, sulfur concrete.

Introduction

Portland cement requires limestone to be heated in kilns at temperatures above 1400 ° C for several hours to form clinker materials. Because fossil fuels are burned to burn kilns and emit stoichiometric carbon dioxide as limestone is converted to calcium oxide, this process generates about one ton of carbon dioxide for every ton of cement and accounts for 5% of global anthropogenic CO_2 production. Unlike traditional cement production, sulfur-based cement production does not rely on high energy or direct carbon dioxide emissions. In addition, sulfur is in net surplus globally. Sulfur is the third most abundant chemical element in oil at a concentration of more than 10 wt. %, and its extraction from oil and gas processing is carried out in accordance with environmental restrictions [1]. Thus, a large amount of sulfur is available as a by-product of these processes. In addition, since sulfur itself is an industrial by-product, significant amounts of carbon dioxide emissions can be reduced by using sulfur-based concrete? Sulfur based concrete is a thermoplastic composite of mineral fillers and sulfur. Early research using elemental sulfur has shown that it has serious durability problems such as repeated freeze and thaw cycles [2].

Therefore, chemical modifiers that polymerize sulfur in order to reduce or eliminate the solid phase transition and thus increase the durability of sulfur-based concrete have been previously studied [3]. This modified sulfur concrete is called polymer sulfur concrete (PSB). It is used as a building material due to its excellent resistance to acidic and saline environments. It is also known that this binder effectively stabilizes / hardens contaminated soils [4]. Unlike conventional hydraulic cement concretes, PSB does not require water and can reach full strength in a few days, compared to 28 days for conventional Portland cement based concretes [2].

The work describes the development and characteristics of a new modified PSB, developed at the Tashkent Scientific Research Institute of Chemical Technology (TNIIKhT) [7]. Instead of an expensive organic modifier, the method uses an industrial by-product crotonic fraction (60-65% crotonaldehyde, and the rest are other aldehydes) as a sulfur modifier. Along with this modifier, other waste, fly ash (for example, ash disposal of the Angren TPP) and sand are used in PSB to provide reactive surface area and as physical fillers. Since most of the main ingredients for the new PSB are industrial by-products (i.e. sulfur, fly ash and croton), this low-cost solution is expected to expand the use of PSB and significantly reduce the environmental impact in the construction sector. This study focused on material characterization of the new PCB. Mechanical and thermal tests, microscopic analysis were performed to assess the suitability of the material for construction [1].

Production of PSB includes preliminary treatment of filler materials (fly ash of Angren TPP and fine-grained quartz aggregate) with croton fraction followed by treatment with elemental sulfur to form a solution of polymerized sulfur. The following mixing ratio was chosen: 54 wt. % sand, 18 wt. % fly ash, 26 wt. % fines quartz and 2 wt. % organic modifier (Table 1). In the study, fly ash is used in conventional cement concrete for its pozzolanic reaction, which reduces the product's carbon footprint in PSB (along with sand) to provide potential reaction sites for polymerization and as a filler component in composite material. The addition of fly ash to PSB is beneficial in increasing the consistency and workability of the mix due to its round shape and suitably sized filler. In the pretreatment step, the filler materials and the organic modifier were mixed and heated to a temperature of 170 - 180 ° C for 12 hours. The materials were combined with elemental sulfur and processed through a 1 mm cross-mixer mill to reduce particle size. Then the mixture was heated and stirred in the molten state at 140-160 ° C for 4-6 hours and poured into molds for cooling (Fig. 1). The average density of the solution samples was 2282 (± 41) кг/м^3 [1].

Fig. 1. Sulfur concrete based on PSB.

able 1 Polymer sulfur concrete mixture recipe.

Additives	Sulfur	Sand	Fly ash	Organic modifier	Total
wt. %	26	54	18	2	100

The thermal properties of elemental sulfur and PSB were measured using differential scanning calorimetry (DSC, PerkinElmer DSC 6000). For this test, a 30 mg sample was loaded into an aluminum crucible and heated at a rate of 5 ° C / min in a controlled environment with flowing inert nitrogen gas. The temperature range was chosen from 25 to 200 ° C. Since there were no significant temperature fluctuations below 90 ° C, the temperature range of 90-180 ° C was used in both samples, as shown in the DSC thermogram in Fig. 2 [5].

The amount of unsaturated hydrocarbons in the croton fraction was determined according to GOST 5475-69 [5]. An iodine value of 51.9 ± 4.7 was obtained. The higher the iodine number, the higher the percentage of unsaturated hydrocarbons available for reaction with sulfur. These data may suggest the possibility of using croton fraction for sulfur polymerization and can be used to evaluate or compare characteristics with other chemical activators.

The stable form of sulfur at ambient temperature is rhombic (S_α), and other main allotropies are monoclinic (S_β) and polymer sulfur (S_∞) [6]. Sα and S_β are composed of S_8 rings, while S_∞ has long chains up to 10^6 atoms long [5].

In addition, S_λ refers to the state of sulfur below 159 ° C, which consists of S_8 molecules, while S_μ denotes an equilibrium mixture of S_∞ and S_8 molecules that can be obtained at temperatures above 159 ° C. In this study, DSC was used to study the thermal behavior (i.e., the phase transition of the allotropic form of sulfur) of both elemental sulfur and PSB. In fig. 3.2 shows the results of the DSC analyzes. In the case of elemental sulfur (red line), the first peak at 108.9 ° C indicates a transition from S_α to S_β. The second main peak at 118.9 ° C and the last peak at 165 ° C show the transition from S_β to S_γ and the transition from S_γ to S_μ, respectively. This proves the presence of polymer sulfur in PSB. A significant decrease in the first peak indicates that the solid phase transition is suppressed due to sulfur polymerization [2]. In addition, the S_α melting observed by DSC occurred at a lower temperature (102.0 ° C) than in the case of elemental sulfur (108.9 ° C). The lower intensity of the third peak may be additional evidence of polymerization or an indication of a small amount of unreacted sulfur present in the PSB sample. The slow, continuous increase in the thermogram for PSB is probably due to an unknown artifact that was in the Al crucible. As mentioned above, appropriately modified sulfur concrete does not undergo allotropic transformation during hardening [1]. Consequently, it has less cooling shrinkage. The current DSC experiment confirms that the unstable solid phase transition at 108.9 ° C appears in elemental sulfur. On the contrary, in PSB endotherms for the S_α transition are almost not found.

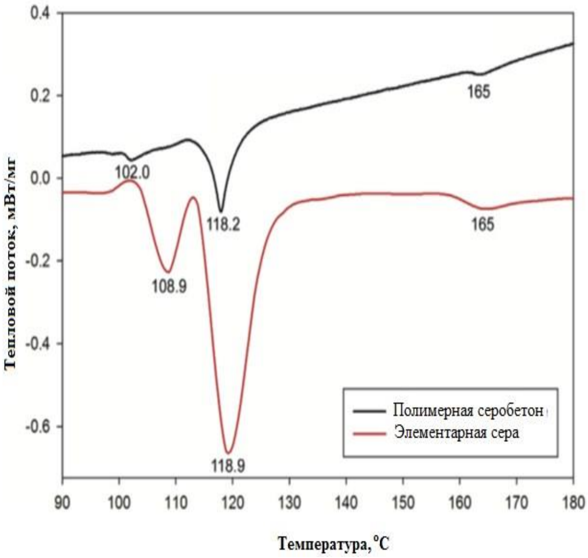

Fig. 2. Thermal properties of elemental sulfur and polymer sulfur concrete.

Table 2 shows the results of mechanical compression and bending tests. The average compressive strength for the six repetitive specimens is 62.3 ± 3.8 MPa and 59.6 ± 4.5 MPa at ambient temperature (20 °C) and elevated temperature (50 °C), respectively. The measured strength is higher than the requirements for most building applications and significantly higher than that of typical hydraulic cement slurries. No significant deterioration in strength was observed at elevated temperature [1].

Table 2
Results of mechanical tests of PSB.

Temperature	20 ºC	50 ºC
Compressive strength of PSB (MPa)	62,3 (3,8)	59,6 (4,5)
Bending strength of PSB (MPa)	8,1 (1,1)	8,0 (2,0)

In this study, a new PSB was characterized using industrial sulfur by-products, fly ash as well as croton fraction and fines. Since the developed PSB is based on an inexpensive chemical modifier (that is, it is also a by-product of the JSC Navoiazot plant), the PSB-based material can be a practical and cost-effective solution for a sustainable building material [5].

It was confirmed that the polymerization of sulfur caused by the croton fraction at high temperature has a satisfactory binding ability to retain fly ash and fines. The measured mechanical properties have shown improved properties such for most building applications. DSC experiments with elemental sulfur and PSB indicate a decrease in the endothermic S_α reaction and therefore a successful polymerization reaction [1; 5].

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