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ISPITIVANJE MORFOLOGIJE SUMPOR-POLIMERNOG KOMPOZITA

MORPHOLOGY INVESTIGATION OF SULFUR-POLYMER COMPOSITE

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Abstract

This research evaluates the effects of the acid environment on sulfur-polymer composite. For this purpose, a morphology investigation consisting of mathematical morphology analysis and SEM analysis was performed. Based on the obtained results, insignificant changes in the pore system of sulfur-polymer composite in terms of deterioration, pore distribution, and homogeneity were observed. Namely, no signs of severe damage were detected at the surface and inside the samples. With increasing the acid exposure time, the material exhibited further changes in the direction of homogenization and drowning of the aggregate in the sulfur binder compared with the starting structure. These facts indicate that sulfur-polymer composite can be effectively used as a construction material in an acid environment.

Key words: *sulfur-polymer composite; mathematical morphological analysis; SEM*

Apstrakt

U ovom istraživanju procenjuju se efekti kisele sredine na sumpor-polimerni kompozit. U tu svrhu izvršeno je morfološko ispitivanje koje se sastojalo od matematičke morfološke analize i SEM analize. Na osnovu dobijenih rezultata, uočene su neznatne promene u sistemu pora kompozita sumpor-polimer u pogledu propadanja, raspodele pora i homogenosti. Naime, na površini i unutar uzoraka nisu otkriveni znaci ozbiljnih oštećenja. Sa povećanjem vremena izlaganja kiselini, materijal se dalje menjao u smislu homogenizacije i utapanja agregata u sumporno vezivo u poređenju sa početnom strukturom. Ove činjenice ukazuju da se sumpor-polimer kompozit može efikasno koristiti kao građevinski materijal u kiseloj sredini.

Ključne reči: *sumpor-polimerni kompozit; matematička morfološka analiza; SEM*

1 Introduction

Sulfur- polymer matrix composites are high performance environmentally sustainable and durable thermoplastic materials made of mineral aggregates, filler and modified sulfur binder which replaces cement and water in conventional Portland cement composite at temperatures above the hardening point of sulfur (120 °C) [1]. Using sulfur to make modified sulfur binder is based on its physico-chemical characteristics. Different types of modified sulfur have been created in order to prevent failure of composite material with elemental sulfur as a binder. For this purpose, various chemical modifiers were used to polymerize sulfur and one of them is dicyclopentadiene which reacts with elemental sulfur to form long-chain polymeric polysulfides [2]. Unlike conventional Portland cement- based composites, sulfur- polymer composites are produced without water and achieve final strength in a few days. In spite of durability and sustainability of sulfur- polymer matrix composites, their wide use is limited due to the high price of chemical modifiers.

According to our own terminology, the term *modified sulfur binder* means a mixture of elemental sulfur and modified sulfur-sulfur polymer [3]. Recent experience all over the world shows that composite materials produced with modified sulfur binder instead of cement and water have significant chemical and physico-mechanical advantages comparing with Portland cement composites.

Since all materials during their service life are exposed to different external impacts that cause certain kind of reaction, the idea of this research was to examine the quality of sulfur- polymer composite in extreme conditions, during the accelerated destruction testing in hydrochloric acid solution.

Image analysis of microphotographs taken through a microscope was applied as a conventional way of these examinations, whereby the samples were cut and changes of the obtained slices were quantified. The question of the resolution necessary for observing the surface or the interior of the samples in this case was solved by using two types of microscopes, optical and scanning electron microscope (SEM).

2 Materials and Methods

2.1 Samples preparation

Sulfur-polymer composite samples were fabricated according to the manufacturing technological procedure described in [1,4]. The process included mixing both melted sulfur and modified sulfur into heated and homogenized dry mixture of aggregate and filler at sulfur melting temperature, 132–141 °C. After homogenization and mixing, sulfur-polymer composite mixture was casted into molds preheated at 120 °C and vibrated for 10 s on a vibrating table. The surface of each sample was finished and left to harden inside the molds, at room temperature. The samples were removed from the mold after 3 h and cured at room temperature for 24 h.

2.2 Accelerated destruction

In order to evaluate quality of sulfur- polymer composite, cube samples were tested under immersion in 10 % HCl solution for 7, 14, 21, 60 and 180 days.

2.3 Morphology investigation

The resulting damage of sulfur-polymer composite was assessed by monitoring and analyzing morfology changes.

Microstucture changes of sulfur-polymer composite samples during the accelerated destruction agent influence were observed using optical and scanning electron microscope (SEM).

Slices, that is cross-sections of the samples, were taken by the optical stereomicroscope with CCD (Charge Coupled Device) camera, LEICA DC 150 in order to analyze the volume changes. The samples of sulfur-polymer matrix composite were cut into four slices by diamond cutting blade that is used for the preparation of mineralogical specimens. Software Image Pro Plus, version 6.2, with the appropriate programming procedure was used and mathematical morphological analysis was performed for the analysis of the obtained images [5].

In order to analyze the morphological and structural changes, sulfur-polymer composite samples were taken by scanning electron microscope JEOL JSM 5800, Vega Tescan TS 5130MM.

3 RESULTS AND DISCUSSION

3.1 Mathematical Morphological Analysis

Set of selected morphological parameters served for the quantification of changes in the analyzed samples. Cumulative analysis of the measured parameters changes enabled the quantification of the accelerated destruction agent effect on the material quality, that is, on the structure properties.

The surfaces of the slices obtained by cutting samples of sulfur- polymer composite are taken by a stereomicroscope, and typical images are shown in Figure 1.

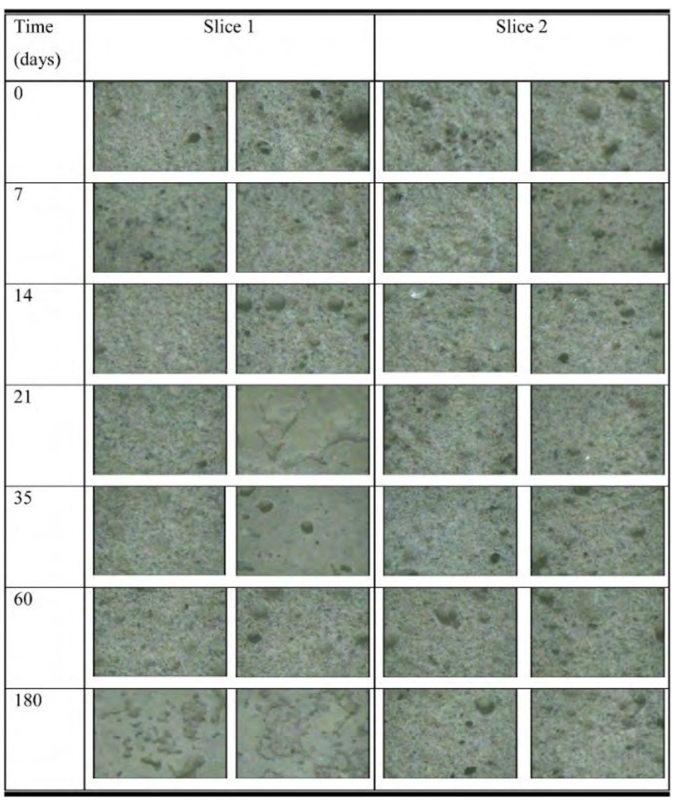

Figure 1. The stereomicroscope images of sulfur- polymer matrix composite samples during accelerated destruction testing.

After data reduction, extensive mathematical analysis using statistical procedures was performed for each sulfur- polymer composite sample as well as time analysis of all measured parameters. Statistical analysis was performed to define the homogeneity of the sample. It was important that the values of measured parameters on the slices of a certain sample do not vary significantly. It means that the inhomogeneity shows whether a sample is not affected by the influence of the agent of accelerated destruction throughout the volume, or does not respond.

Mean values of measured parameters during the treatment time, Figure 2, indicate that there is a homogeneity change, meaning that more significant interactions took place than in the first 21 days. In this way, the impact of sulfur on the material behavior in terms of inhibition of the observed changes in the structure can be discussed.

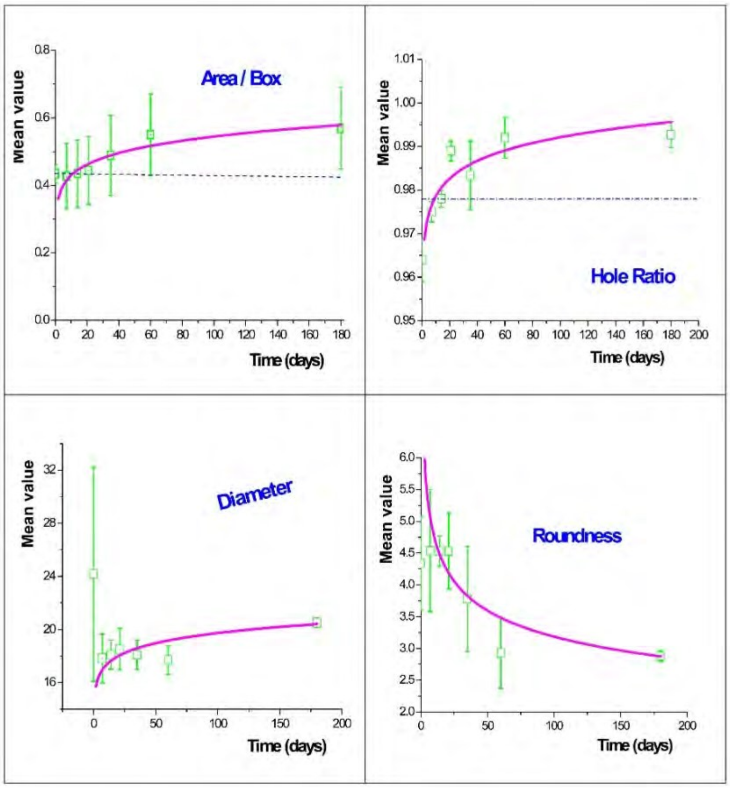

Figure 2. Mean values of measured parameters- homogeneity change of sulfur- polymer composite during accelerated destruction testing.

Based on the given analysis, it may be concluded that the greatest homogeneity increase takes place at 21st day of treatment. The morphological analysis is interesting after 21 days of treatment because of evident greater volume changes of certain parameters which positively or negatively affect the homogeneity achieved at approximately 21st day of accelerated destruction agent influence. However, regardless the effect, the action of the accelerated destruction agent was towards rearranging sulfur, which was the main moderator of homogeneity changes.

Further analysis was the analysis of time changes in cumulative parameters for each sample.

Figure 3 shows the graphs for four measured parameters through the volume of sulfur- polymer composite sample during the treatment time from 0 to 180 days.

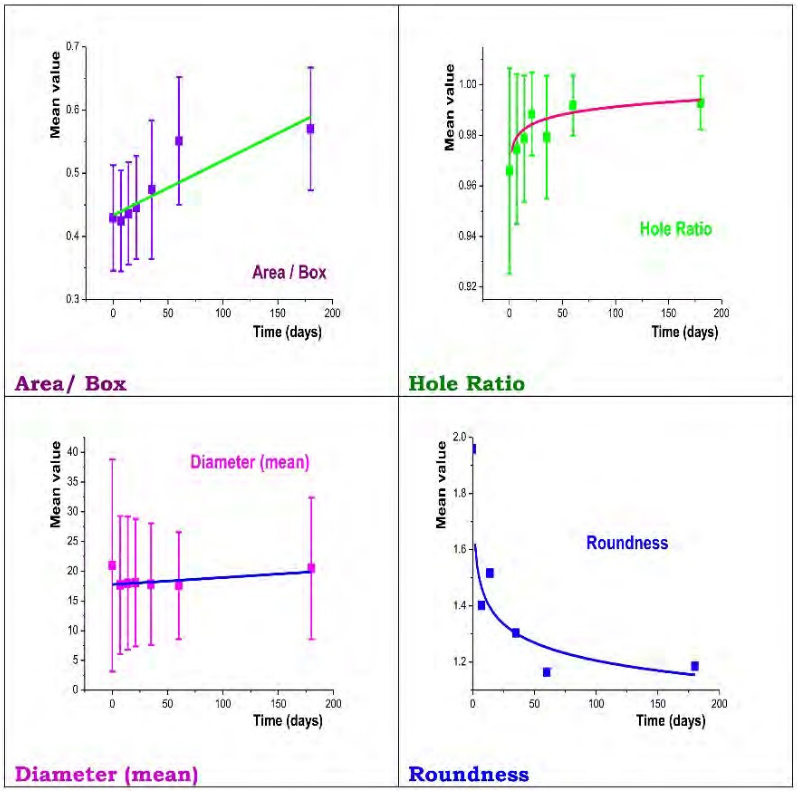

Figure 3. Change of measured parameters through the volume of sulfur- polymer matrix composite samples during accelerated destruction testing from 0 to 180 days.

As it can be seen from the graph shown in Figure 3, the time change kept the same form- power function. Compared with the surface analysis, a lower correlation degree is present here. This is entirely in accordance with the supposed model, since, unlike surface changes, bulk changes are cumulative changes of higher interaction material- agent, which results in a lower correlation degree between the examined parameters.

3.2 Morphological Analysis by SEM

The testing methodology included monitoring the structure changes of sulfur- polymer composite before the treatment and up to six months of the accelerated destruction agent action.

Microphotographs generated by the SE detector were analyzed in order to determine the morphological structure of the samples. To determine the final distribution of sulfur in the structure, element mapping, which means observing a sample in the energy range of the selected element, was applied.

SE microphotographs of sulfur- polymer composite samples are presented in Figure 4.

As seen on SE microphotographs, the existing binding phase, sulfur, and separated aggregate grains can be distinguished.

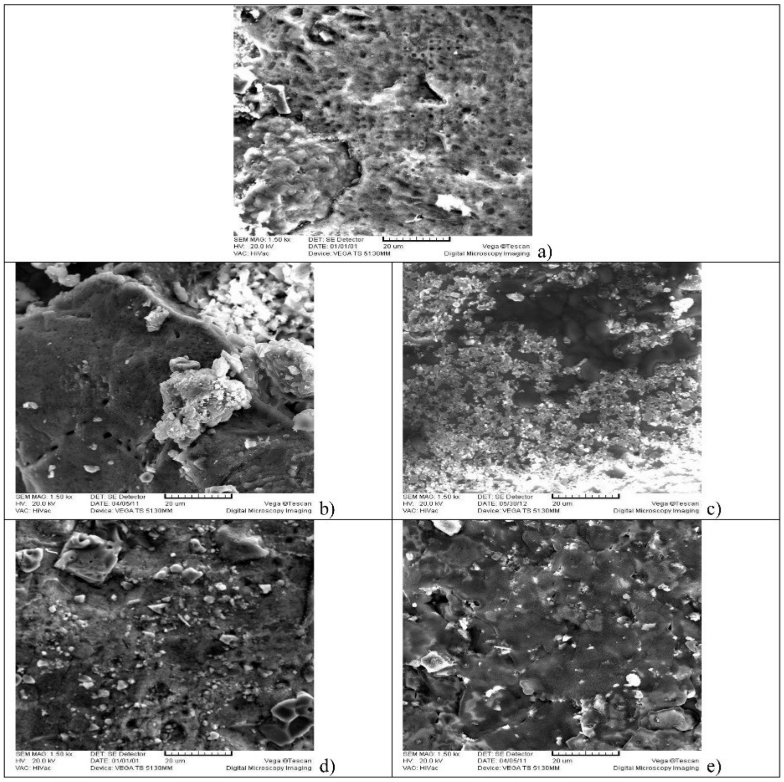

Figure 4. SE microphotographs of sulfur- polymer matrix composite samples during accelerated destruction testing: a) 0 days, b) 14 days, c) 21 days, d) 60 days, and e) 180 days.

Clearly defined aggregate and sulfur binder that looks like a monolithic integral part of the system can be noticed in the structure of the untreated sulfur- polymer composite sample, Figure 4 a).

Sulfur- polymer composite samples exposed to accelerated destruction show more pronounced monolithic structure. Also, ‘‘drowning’’ of the aggregate into the binder phase- sulfur, homogenization, can be observed. With increasing the treatment time, the structure exhibits further changes in the direction of homogenization and drowning of the aggregate in the sulfur binder compared with the initial structure, Figure 4 b), c), d). Based on the analysis of the structural changes, secondary homogenization of sulfur- polymer composite treated by acid, with clearly visible secondary binding of the aggregate and primary binder phase, expressed after 180 days, is evident, Figure 4 e).

4 Conclusion

According to the trends of selected parameters variations throughout the samples volume, it was concluded that a homogenization of sulfur-polymer composite sample occurred during the time of accelerated destruction. The greatest homogeneity increase happened at 21st day of treatment. The influence of the accelerated destruction agent was towards rearranging sulfur, which was the main factor of homogeneity changes.

It was possible to show sulfur- polymer composite structure rearranging by observing the material structure with higher resolution and by choosing another method that would indicate the volume changes. The scanning electron microscopy was applied as a highly resolution method. This method clearly identified changes- rearrangement of the structure during the time.

According to the presented research, it can be concluded the accelerated destruction can be used to provoke changes in the structure of materials and thus properties changes. The used methods adequately demonstrate the possibility of detecting destruction influence on the sulfur- polymer composite structures.

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