



# COMPATIBILITY OF SHUNGISITE MICROFILLERS WITH POLYCARBOXYLATE ADMIXTURES IN CEMENT COMPOSITIONS

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## ABSTRACT

The problem of the development of new multi-component binders is relevant in the present time. Ground rocks and by-products of the industry can be used in multi-component binders for various applications. Mineral additive based on shungite rocks is used for protective concrete against ionizing radiation. Shungite rocks differ in genesis, mineral composition as well as carbon and silica content. The efficacy of aggregates based on shungite rocks and shungisite in cement concrete as protective material against ionising radiation is known. However, influence of shungisitemicrofillers on the properties of cement compositions with polycarboxylate superplasticizers was not investigated. Morphology, element analysis of shungisite particles as well as compatibility of shungisitemicrofillers with polycarboxylate superplasticizer in cement compositions have studied in the paper. Shungisitemicrofillers were obtained under heating shungite rock at the temperature of 1000°C and its subsequent grinding. It has stated that the water demand of cement paste with normal consistency had increased with the increase of the fineness of the shungisitemicrofiller. The plasticizing effect of polycarboxylate-based superplasticizer has depended on the composition of shungisite. The data of determining the electrokinetic properties of the shungisite micro-particles have confirmed this. Results can contribute to the rational use of shungisitemicrofillers in cement composite to create effective protective materials with shielding properties against electromagnetic radiation or cement composites with electro-conductive properties.

**Keywords:** shungite rock, shungisite, microfiller, protective cement composite, superplasticizer.

## 1. INTRODUCTION

Shungite rock is the carbon-containing rock that is intermediate form between the amorphous carbon and the crystal graphite containing carbon up to 30 %, silica up to 45 % and silicate mica up to 20 % [1]. Shungite rock is the fossilized carbon material of sea bottom Precambrian sediments with high degree of carbonization.

Shungite rocks have mostly the amorphous structure [2]. Carbon of shungite rocks has the specific graphene-like structure [2] and it is consider as special natural allotropic modification of carbon which is called the shungite carbon. The shungite carbon has the heterogeneous molecular structure in which carbon occurs as 10 nm sized globules. These globules are non-uniform distributed [3].

The unusual physico-chemical and structural properties of shungite rocks are used in diverse industrial and environmental applications including metallurgy, water purification, thermolysis and organosynthesis of cyclic hydrocarbons. Shungite rock is effective sorbent for removal of organic and inorganic substances, pathogenic bacteria and heavy metals from contaminated water [3].

Shungite-bearing rocks have been classified according to their carbon content in paper [4]. According to this classification one can define the following five types of shungite: shungite - I (80-100 wt.% C), shungite - II (35-80 wt.% C), shungite - III (20-35 wt. %C), shungite - IV (10-20 wt.% C) and shungite - V ( < 10% wt.% C). This classification is suitable for shungite-bearing rocks since they may have different compositions and lithologies. For example, shungite-V may contain tuff,

dolostone, limestone, basalt or gabbro. Types I, II and III contain less SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> quantity.

Shungite rock is characterised by low specific gravity 1.7-1.9 g/cm<sup>3</sup>. The heated shungite rock is named shungisite and it has the specific gravity in the range 0.35-0.50 g/cm<sup>3</sup> after heated at the temperature of 1000°C.

The efficacy of the protective composition of the cement concrete with shungisite dust and shungisite sand against ionising radiation has been shown in papers [5,6]. The reason of grate efficacy is in the presence of the large number of nano-pores in the shungisite particles that reduce fracture of concrete under exposure of ionizing radiation. However, there are no published data about the morphology and composition of shungisitemicrofillers, about influence of shungisitemicrofillers on the properties of fresh concrete with superplasticizer.

Shungisitemicrofiller can be the effective additive for protective cement composites against ionising radiation. Modern cement composites are produced using supplementary components and superplasticizers [7-14]. The problem of compatibility of mineral fillers with superplasticizers to enhance the workability of fresh concrete is studied in papers [15-18]. The fluidity of plasticized cement pastes depends on fineness of cements and additives [17-20], on quantity of modifiers [19,20]. The increase of plasticizing effect of the superplasticizers with fillers based on ground quartz sand and slag are stated in the papers [21-23]. Superplasticizer is used in concrete to improve workability of fresh concrete and enhance properties of hardened concrete [21,24]. However, the interaction of mineral additives with the



superplasticizer is still under study by many researchers [25-30]. Superplasticizers are mostly negatively charged (anionic) polymers that tend to adsorb on the positively charged (cationic) hydration compounds. This adsorption reduces the positive charge of particle surface or inverts it. However, due to the presence of high amounts of cations and anions the surface effects can be more complicated. The dispersion of particles is induced by electrostatic repulsion or steric hindrance [25-30]. The adsorption of superplasticizers mostly depends on their type as well as the mineral particle electrokinetic properties.

The zeta-potential can be an indicator for the degree of repulsion between the charged particles in a cementitious system [23,30-33] and it allows estimate the adsorption of the SP onto the solid surfaces of the hydrates. Superplasticizer adsorption is affected by the

zeta-potential and it means that at more positive zeta-potential the molecules of superplasticizer are more likely to be adsorbed onto the particle surfaces in cement suspensions [30]. However, adsorption depends on ionic pore solution of fresh cement paste.

The aim of the paper is to study the influence of fineness and morphology of shungite particles on normal consistency of cement paste, on plasticizing effect of polycarboxylate-based superplasticizers as well as electrokinetic properties of shungite particles.

## 2. MATERIALS AND METHODS

In this research the Ordinary Portland Cement CEM I 42.5N was used. The chemical and mineralogical compositions of Portland cement are presented in Tables-1 and 2.

**Table-1.** Chemical composition of portland cement.

CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	Na <sub>2</sub> O <sub>eqv</sub>	CaO <sub>cb</sub>	L.O.I.
63,80	21,20	4,90	3,90	1,00	2,80	0,60	0,13	0,52	0,30	1,10

**Table-2.** Mineralogical composition of Portland cement.

C <sub>3</sub> S	C <sub>2</sub> S	C <sub>3</sub> A	C <sub>4</sub> AF
63,0	14,7	6,5	13,0

The chemical compositions of two types of shungite rocks from Zazhoginsky deposit (Karelia, Russian Federation) are presented in Table-3.

**Table-3.** The chemical composition of shungite rocks.

Chemical component	Content, % (w/w)	
	shungite rock (S)	shungite rock (C)
C	30.0	34.1
SiO <sub>2</sub>	57.0	52.2
TiO <sub>2</sub>	0.2	0.23
Al <sub>2</sub> O <sub>3</sub>	4.0	3.61
FeO	0.6	0.2
Fe <sub>2</sub> O <sub>3</sub>	1.49	0.84
MgO	1.2	2.67
MnO	0.15	-
CaO	0.3	1.8
Na <sub>2</sub> O	0.2	-
K <sub>2</sub> O	1.5	0.61
S	1.2	0.7
H <sub>2</sub> O	1.7	2.2

The shungite microfillers were produced by heating shungite rocks at the temperature of 1000°C and their grinding. Shungite microfillers were marked as S1, S2, C1, C2 according their fineness. The particle size distributions were obtained using the HELOS/BR analyzer of Sympatec GmbH. The results are presented in Table-4.

The electron microscopic images were obtained by the SEM Supra 55VP-3249 of Zeiss. The element analysis was obtained using the INCA energy dispersion spectrometer.

The z-potential of the shungite particles was investigated in the cement pore solution that extracted from the cement paste with the water-to-cement ratio



(W/C) equal to 0.5. The pore solution and shungisite fillers were mixed at W/C=0.5. Then different dosages of superplasticizer were added by weight of the binder. It makes sure that the ion and superplasticizer adsorption takes place under realistic conditions. The measurement of

the z-potential was conducted using the Zetasizer Nano of Malvern Instruments by electrophoresis method.

The water-reducing polycarboxylate-based admixture Stachement 2000 was chosen and it is marked as PC.

**Table-4.** The particle size distributions of shungisite.

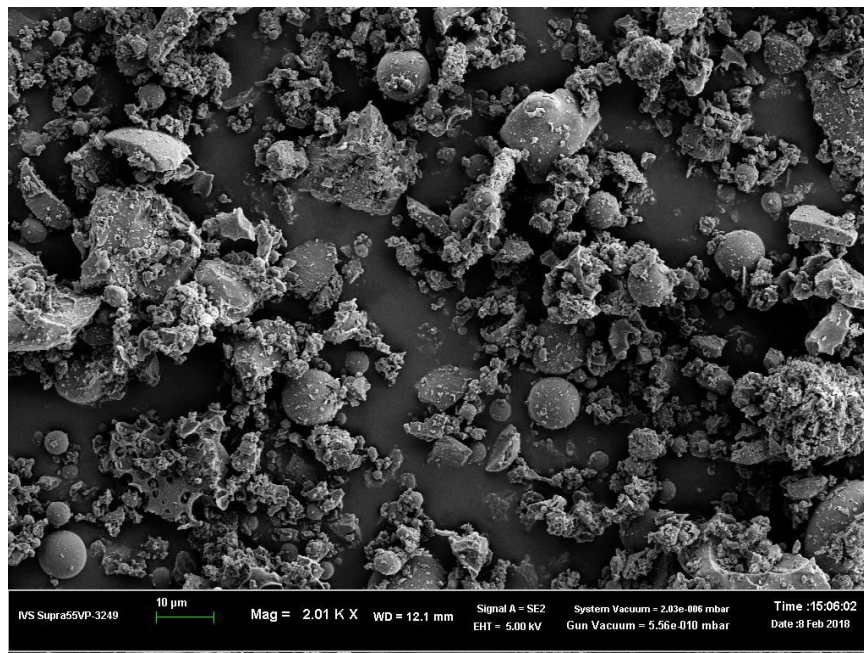
	The quantity of particles with size less then, %				
	1µm	5 µm	10 µm	50 µm	80µm
S1	0.9	6.7	14.5	61.1	86.9
S2	5.3	34.6	46.8	96.9	100.0
C1	1.0	7.5	14.4	58.6	88.7
C2	4.9	37.8	47.6	94.9	100.0

### 3. RESULTS AND DISCUSSIONS

Cement composites containing the coarse shungisite particles with size 100-250µm in amounts up to 30% by weight of OPC provide the shielding ability against ionizing radiation in particular  $\gamma$  - radiation [5]. The disadvantage of these cement composites is the low strength from 25 to 35 MPa. The problem of increasing the composite strength and durability is relevant. The solution can be the use of shungisitemicrofillers with fineness equal to Portland cement fineness. Based on the above-mentioned discussion the quantity of shungisitemicrofillers was chosen up to 30% in this research.

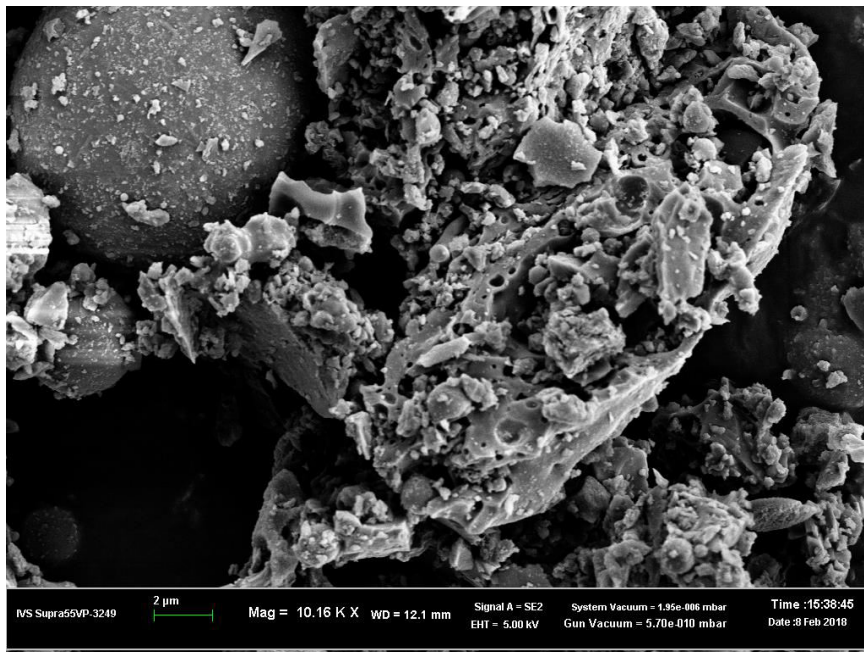
#### 3.1 Morphology of shungisite particles

According to the electron microscopic images one can conclude that shungisite particles differ in morphology. Three types of particles are produced after heating of shungite rocks and grinding: spherical and smooth particles, porous particles and non-porous particles as shown in Figures 1-4. Also the large number of conglomerates of particles one can see that contain mix of all three types of particles: spherical, porous and non-porous particles. These conglomerates have sizes from few microns to tens of microns as shown in Figure-2. The mix of three types of particles one can see inside spherical particles as shown in Figure-4.

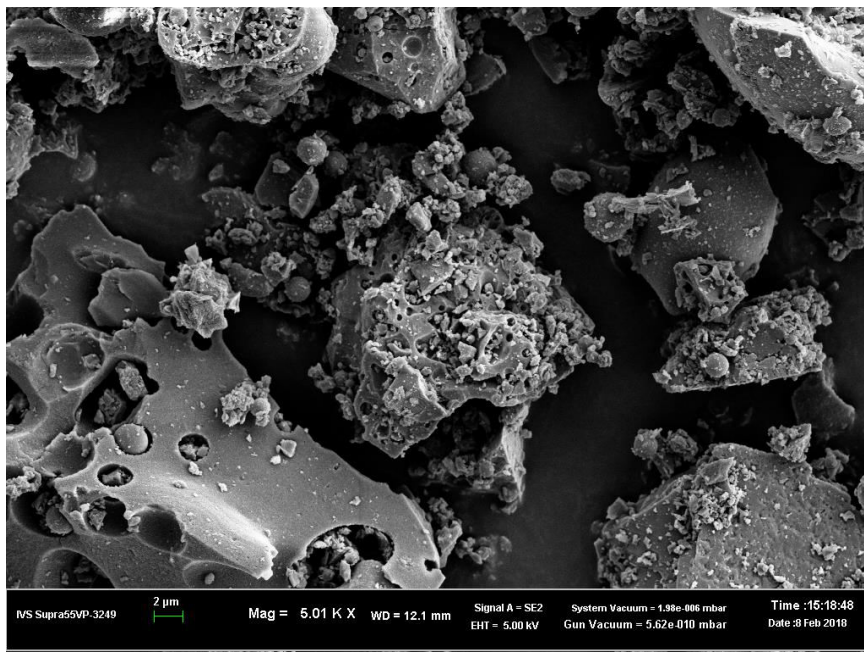


**Figure-1.** Types of shungisitemicrofillers: spherical, porous and non-porous particles.





**Figure-2.**Types of shungisitemicrofillers: spherical and porous particles.



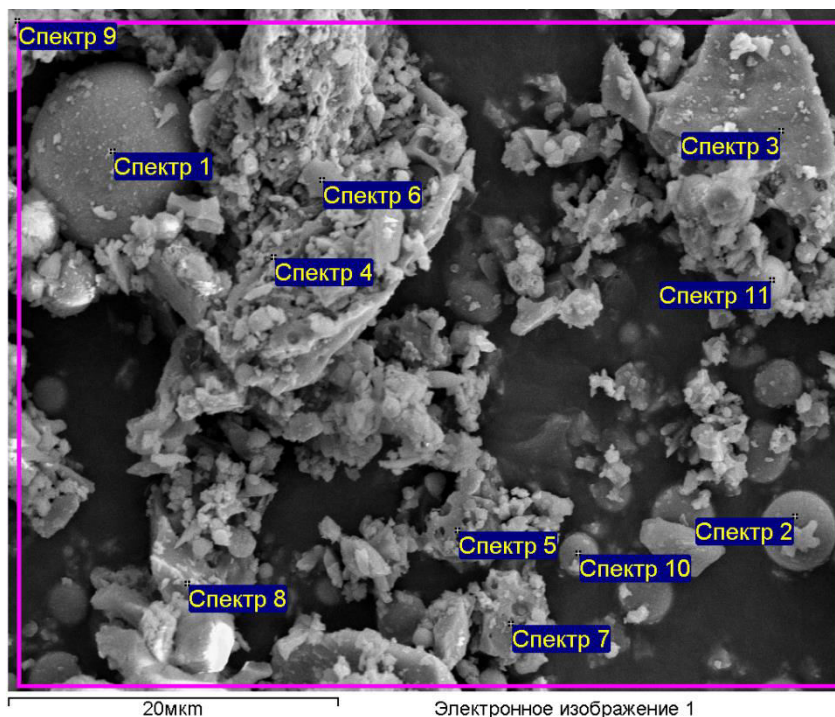
**Figure-3.** Types of shungisitemicrofillers: porous and non-porous particles.



**Figure-4.** Types of shungisitemicrofillers: spherical particles.

One can suppose that the ratio among the three types of shungisite particles depends on the composition of initial shungite rock as well as burning temperature. The dependence of the ratio among the types of

shungisiteparticles from the composition of original shungite rock and burning temperature was not established at this stage of research.



**Figure-5.**Element analysis of shungisite particles.

The element analysis of shungisite particles is presented in Table-5 according data in Figure-5. The results show that all shungisite particles have high silica

and alumina contents. The non-porous particles have high carbon content. However the silica and alumina contents have no correlation with type of particles.

**Table-5.** Element analysis of shungisite particles.

Spectr	C	O	Na	Mg	Al	Si	P	S	K	Ca	Ti	Fe	Total
1	4.71	48.96	0.87	0.48	19.18	18.39	0.09	0.20	1.79	0.57	0.91	3.86	100
2	9.92	39.34	0.71	0.63	9.94	16.06	0.00	0.15	1.46	0.55	0.73	20.51	100
3	18.53	20.59	0.25	0.54	25.78	25.92	0.23	0.12	4.38	0.46	2.20	1.01	100
4	10.27	48.97	0.45	0.27	15.63	21.41	0.00	0.00	1.48	0.00	0.48	1.05	100
5	8.09	47.47	0.41	0.57	16.04	21.57	0.00	0.12	2.25	0.23	0.41	2.85	100
6	50.64	31.62	0.23	0.17	6.62	8.05	0.00	0.16	0.57	0.19	0.48	1.26	100
7	9.29	48.66	0.44	0.55	14.94	20.68	0.00	0.11	2.62	0.27	0.22	2.22	100
8	34.83	16.42	0.27	0.39	15.73	25.50	0.00	0.23	4.04	0.93	0.50	1.16	100
9	44.92	31.41	0.44	0.35	6.28	11.22	0.00	0.23	1.23	0.83	0.46	2.64	100
10	22.12	34.50	0.12	7.22	0.62	1.98	13.15	0.23	0.12	15.55	0.00	4.38	100
11	12.17	40.92	1.12	0.57	15.54	19.71	0.20	0.09	2.91	0.33	0.21	6.23	100
Ave- rage	20.50	37.17	0.48	1.07	13.30	17.32	1.24	0.15	2.08	1.81	0.60	4.29	100
Stan- dard de via- tion	15.91	11.45	0.30	2.05	6.95	7.44	3.95	0.07	1.33	4.57	0.58	5.62	
Max	50.64	48.97	1.12	7.22	25.78	25.92	13.15	0.23	4.38	15.55	2.20	20.51	
Min	4.71	16.42	0.12	0.17	0.62	1.98	0.00	0.00	0.12	0.00	0.00	1.01	

Therefore the micro-particles of shungisite additive have complex morphology. All micro-particles have high silicon and aluminium contents that can be up to 25.9% and 25.7% accordingly. Also there are such elements as carbon, titanium, calcium, potassium, sodium, iron etc. in shungisitemicrofiller compositions. The micro-particles of shungisite additive have greater quantity of nano-sized pores as shown in Figures 2 and 3. It can be useful in cement based protective concrete against

ionizing radiation since the nano-sized pores can be additional space for accumulation of the water hydrolysis products including gases.

## 2.2 Normal consistency of the cement pastes with shungisitemicrofiller

The changes of the normal consistency of cement pastes with shungisitemicrofiller of different fineness (according to Vicat method) is presented in Table-6.

**Table-6.** The normal consistency of cement pastes.

Type of microfiller	Microfiller quantity, % by weight of OPC	Water demand of cement pastes of normal consistency, %
No microfiller	0	25.4
S1	30	28.2
S2	30	35.3
C1	30	27.5
C2	30	33.7

The normal consistency of cement pastes increases with the increase of the shungisitemicrofiller fineness. This can decline the workability or increase the capillary porosity that is the reason of the concrete durability reduction. Superplasticizers enhance the workability or reduce the water demand of cementitious paste. Therefore, the interaction of polycarboxylate admixtures with shungisite additives is necessary to study.

## 2.3 Water-reducing effect of polycarboxylate admixture in shungisite-Portland cement compositions

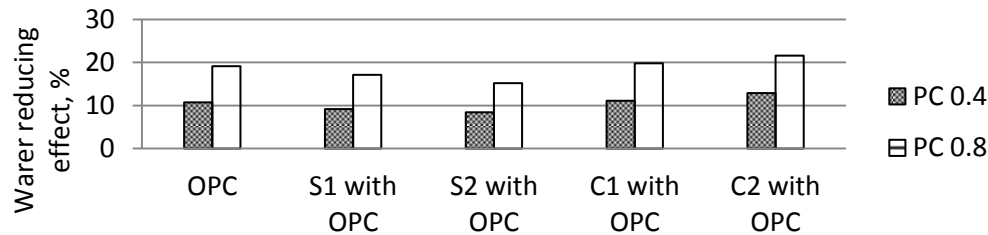
The water-reducing effect of polycarboxylate admixture in shungisite-Portland cement compositions compared to the cement paste without filler was studied and shown in Figure-6. The amount of shungisitemicrofiller was 30%. The amounts of superplasticizer were 0.4 and 0.8 % by weight of total





binder. The water-reducing effect of admixture in compositions with equal fluidity was defined in per cent as the reduction of water demand of plasticized cement paste

from the water demand of non-plasticized cement paste of normal consistency according to Vicat method.



**Figure-6.** The water-reducing effect of the polycarboxylate admixture.

The reduction and increase of the water-reducing effect of polycarboxylate admixture depend on the microfiller type and its fineness as shown in Figure-6. There is the problem of compatibility of admixture and shungisitemicrofiller. This problem was discussed for the others mineral additives and Portland cement in papers [19,22,23,34,35,36]. According to Figure-6 the microfiller C2 has the greatest increase of water-reducing effect of the polycarboxylate admixture. The water-reducing effect decreases in cement pastes with S1 and S2 shungisitemicrofillers and especially with the fineness increase. On the contrary, the water-reducing effect of the polycarboxylate admixture increases in cement pastes with C1 and C2 shungisitemicrofillers and especially with the fineness increase. Therefore the polycarboxylate-based admixture interacts differently with shungisite particles

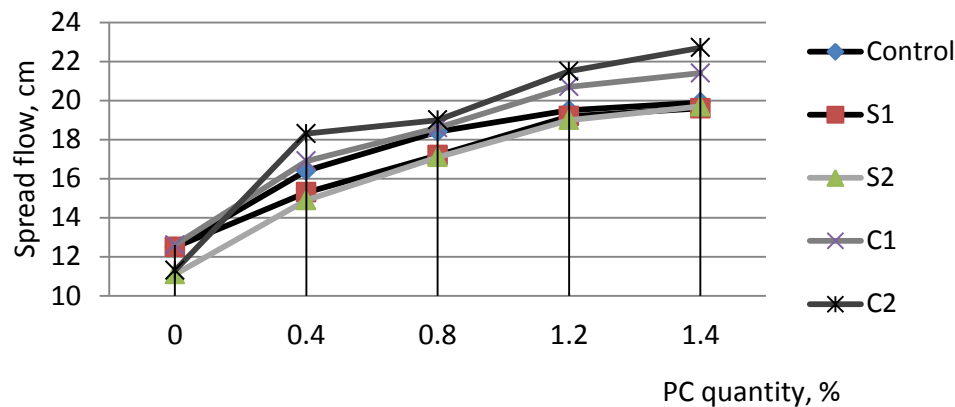
and the problem of compatibility of admixture and shungisitemicrofiller should be studied.

## 2.4 Plasticizing effect of polycarboxylate admixture in the shungisite-cement fresh mortars

In order to observe the fresh mortar properties with shungisitemicrofiller and polycarboxylate admixture, the mini slump flow test was done on the fresh mortars using the Hagman cone. The slump flow was measured in 15 minutes from the time of introduction of water (with and without admixture). The optimum dosage of admixture was observed and the point of segregation was noted. The abbreviation of the mixes and dosages of admixture are presented in Table-7. All tests were conducted at 20°C. The spread flows of fresh mortars depending on admixture quantity and the shungisite filler fineness are shown in Figure-7.

**Table-7.** The mixture compositions.

The abbreviation	Type of microfiller	The ratio of microfiller-to-OPC, % by weight of Portland cement
control	No microfiller	0/100
S1	shungisite S1	30/70
S2	shungisite S2	30/70
C1	shungisite C1	30/70
C2	shungisite C2	30/70



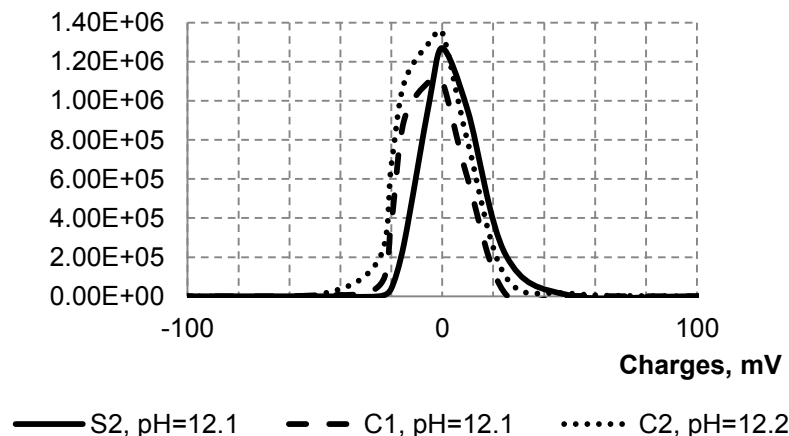
**Figure-7.** Spread flow of mortars depending on admixture quantity and shungisite filler fineness.

The optimum dosage of admixture was up to 1.2% as the point of segregation was noted at higher admixture quantity. The data of the spread flows are correlated with the data of paragraph 2.3. The plasticizing action of the admixture increases in cement pastes containing C1 and C2 microfillers and especially with the increase of grinding fineness. The spread flow of plasticized fresh mortar containing shungisitemicrofiller in quantity of 30% from OPC with particles size less 50  $\mu\text{m}$  in quantity of 95% has increased up to 15%.

### 2.5 Electrokinetic properties of the shungisite microfiller particles

The results [23, 30, 32] have shown that polycarboxylate-based admixtures are strongly adsorbed by positively charged materials. So the measurements of z-potentials of microfiller particles make it possible to define their ability to adsorb anionic plasticizing admixtures.

One can see that the negatively and positively charged active centres exist on the shungisite micro-particle surfaces as shown in Figure-8. The greatest number of negatively charged active centres is located on the C1 and C2 microfillers surfaces and the number of negative centres grows with the microfiller fineness increasing as in the case of C2 microfiller.



**Figure-8.** Quantitative distribution of charges on the shungisite micro-particle surfaces.

The measurements of z-potential of shungisitemicrofillers give some new information on the ability of these particles to influence on the plasticizing effect of the superplasticizers in the shungisite-cement compositions. The studies have showed that particles of the S2 microfiller have positive z-potential at pH=12.1; particles of the C1 microfiller have negative z-potential. The z-potential of the C2 microfiller particles has been more negative than the z-potential of the C1 microfiller

particles because of the smaller sizes of C2 microfiller particles. The ion concentration of the pore solution significantly has changed the z-potential. For instance, in the case of the quartz flour as shown in paper [32], the high pH of the artificial pore solution caused an increase of negative surface sites ( $\text{SiO}^-$ ).

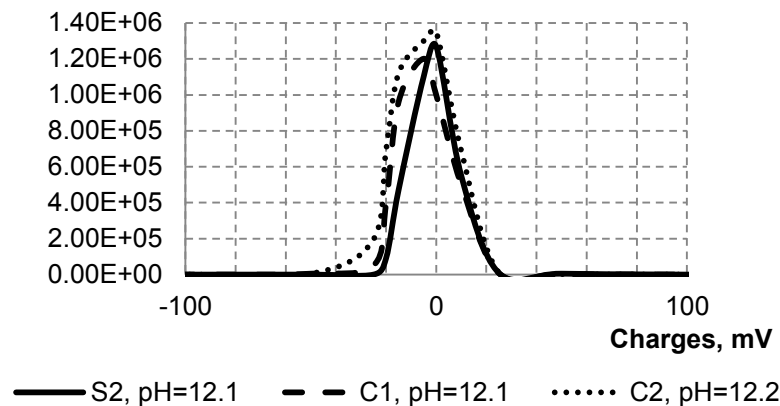
In this study the addition of the PC to the suspensions was done in 5 minutes from the time of introduction of water to allow formation of the first





hydrates. Measurement of the z-potential for all suspensions was done in 15 minutes from the time of introduction of water to allow interaction between the PC

and the ions in the suspension. One can see that the greater number of negatively charged active centres exist on the microfiller particle surfaces as shown in Figure-9.



**Figure-9.** Quantitative distribution of charges on the shungisite micro-particles surfaces with PC.

Therefore one can assume that electrokinetic properties of shungisitemicrofiller have significant effect on the plasticisation of fresh mortar. Based on the above mentioned results in section 2.4 one can conclude that the major part of PC is spent on dispersing Portland cement particles in suspension with C2 microfiller since PC does not adsorb on C2 microfiller particles. It is confirmed by minor change of the quantitative distribution of charges on the C2 microfiller particle surfaces with PC. The decrease of workability with the S2 microfiller and the improvement of workability with the C2 microfiller are observed compared with the data of Figures 7 and 9.

Thus, the introduction of shungisitemicrofiller can considerably increase the plasticizing effect of PC that provides the improvement of the workability and gives the opportunity to obtain the denser cement matrix with less volume of capillary pores that leads to the concrete durability increase. Also the shungisite micro-particles with unique nano-sized pores structures can be useful in cement based protective material against ionizing radiation since the water hydrolysis products including gases can accumulate in these pores.

### 3. CONCLUSIONS

The efficacy of shungisite coarse dust and fine aggregates in protective cement concrete against ionising radiation is known. However, this concrete has low strength from 25 to 35 MPa and durability. The issue of increasing the strength and durability of such protective concrete is relevant. One of the ways to increase the concrete strength and to ensure the protective properties can be the use of shungisitemicrofiller.

Morphology of shungisitemicrofillers as well as their influence on the water demand and the spread flow of fresh mortars with plasticizing admixture were studied in the paper. The micro-particles of shungisite additive have complex morphology. The micro-particles of shungisite additive have an extensive network of nano-sized pores.

Three types of shungisite micro-particles were obtained after heating of shungite rocks at temperature of 1000°C and grinding: spherical particles, porous and non-porous particles. There was great quantity of conglomerates containing mixes of these types of particles. All micro-particles have high silicon and aluminium contents that can be up to 25.9% and 25.7% accordingly. Also there are such elements as carbon, titanium, calcium, potassium, sodium, iron etc. in shungisitemicrofiller compositions.

It was stated that the water demand of cement paste with normal consistency increases from 25% to 35% with the increase of shungisitemicrofiller fineness. Shungisitemicrofillers were added in quantity of 30% of Portland cement mass. The compatibility of shungisitemicrofillers with polycarboxylate based admixture in cement compositions was studied using assessment of the plasticizing and water-reducing effects of the admixture in cement paste, the spread flow of fresh mortars as well as electrokinetic properties of shungisitemicrofillers. The plasticizing and water-reducing effects of admixture in shungisite-Portland cement paste depend on the composition and quantity of shungisitemicrofiller. The plasticizing effect of admixture decreases in shungisite-Portland cement pastes when shungisite was obtained from initial shungite rock with larger quantity of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  namely 57.0% and 4.0% accordingly. The plasticizing effect of admixture slightly increases in shungisite-Portland cement pastes when shungisite was obtained from initial shungite rock with less quantity of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  namely 52.2% and 3.61% accordingly. This regularity was kept with the increase of grinding fineness of microfillers. The spread flow of plasticized fresh mortar containing shungisitemicrofiller with particles size less 50  $\mu\text{m}$  in quantity of 95% has increased up to 12%. The other kind of shungisitemicrofiller has decreased the spread flow of plasticized fresh mortar up to 10%. The data of determining the electrokinetic properties of the



shungisitemicrofiller particles have confirmed the influence of shungisitemicrofiller properties on the plasticizing and water-reducing effects of polycarboxylate-based admixture.

The results can contribute to the rational use of shungite rocks in cement concrete to create effective protection materials.

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