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Creation of a Cement Stone Resistant To Aggressive Influences of Formation Fluids

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ABSTRACT: A grouting composition based on basalt fiber waste has been developed, which allows to ensure the stability of cement stone in conditions of aggressive action of hydrogen sulfide and salts. We also studied the reducing ability of basalt to water separation of cement slurry and permeability of cement stone. In addition, they provide for a long-time sufficient stability, bending, compressive and tensile strength of a cement stone in an aggressive reservoir environment.

KEY WORDS: rock, clay, drilling, well, fluid, flow, dependence, filtration, experiment, opening, crust, pressure

I. INTRODUCTION

The cement slurry pumped into the annulus between the borehole wall and the outer diameter of the casing string during the transformation of the cement stone and after their formation for a long (sometimes more than 100 years) time has contact with formation fluids and is aggressively affected by them. At each interval of wells where aggressive formation fluids occur - high temperature, highly mineralized water, hydrogen sulfide, carbon dioxide and salt affect the strength of the cement stone in different ways.

In the sections of one area of southwestern Uzbekistan, the Amu Darya and Fergana depressions, all of the above aggressive formation fluids are present. When cementing a column, the prepared plugging mixture of the same composition does not always satisfy the requirements for plugging wells in the presence of various aggressive fluids. When cementing one casing, it is very difficult and expensive to prepare a special grouting composition for each fluid in the intervals.

II. LITERATURE STUDY

Scientists and industrialists of our republic and foreign countries of the world have recommended a number of developments of stable or high or low temperature, or salt or hydrogen sulfide aggression. However, today there is no inexpensive, affordable and reliable one additive that could comprehensively solve a number of problems associated with the separation of layers.

III. EXPERIMENTAL RESULTS

In connection with the above, we carried out experimental studies on the development of the composition of the grouting composition stable, hydrogen sulfide and salt aggression, as well as high reservoir temperature, i.e. to develop the composition of the grouting composition that allows to comprehensively solve several problems associated with the separation of layers.

Waste of basalt fiber and reinforcement from Mega Invest Industrial company located in Forish district of Jizzakh region was used as an additive to cement mortars.

Basalt is an igneous volcanic rock. The plutonic analogue of basalts is Gabbro, and the Hypobysal analogue is dolerites. Basalt varieties include Trapps. Coloring - dark, black, dark gray. Structure: dense, fine-grained. The density of basalt rocks can vary from 2530 to 2970 kg/m³. It should be noted that after the melting of the basalt, its density will decrease to 1410-1400 kg/m³. This advantage allows the cementing of highly permeable, highly porous and fractured rocks.

Hardness on the MOSS scale from 5 to 7. Melting point 1100-1450 °C. The compressive strength of different basalts can be in the range of 60-400 MPa. Basalt has high tensile strength and no less high modulus of elasticity [1].

Such high resistance is its dispersed formation, using the principle of composition, in which the cement matrix provides resistance to compressive stress, and the forming fibrous component (fiber) - to tensile and bending stress.

The chemical composition of basalts is represented by 45-53 % silica up to 7 % alkaline oxides, 14.5-17.9 % aluminum oxides, 7.3-8.1 % iron, 7.1-9.3 % magnesium, 9.1- 10.1 % calcium, as well as oxides of titanium, manganese and lead. There are industrial types of basalts in our republic in the autonomous republic of Karakalpakstan on the territory of Mount Sultanuvsays, Jizzakh region at the eastern end of the Koytash mountains, Samarkand region of Zirabulak mountains and Surkhandarya region of Mount Surkhantau (Badava area).

During the research we used basalt fiber waste. Basalt fiber is an artificial inorganic material obtained from natural basalt by melting at a temperature of 1400 °C. Basalt fiber is used to obtain basalt fiber, reinforcement and other building materials, which are widely used in the construction of residential buildings and industrial facilities. In this regard, there is a multi-ton waste of basalt fiber and reinforcement. Prior to the study, waste basalt fiber and reinforcement were ground in a laboratory ball mill to the size of cement and the resulting basalt product was added to Portland cement in the amount of 2.0; 4.0; 6.0; 8.0 and 10.0 % by weight of cement.

At the beginning, we studied the effect of basalt fiber waste on the water separation of the cement slurry, which has W/C=0.55 in vertical and inclined cylinders. Comparison of the technological characteristics of the cement stone was carried out by adding sand sand, asbestos and marble powder to the mortar in the same amount. The result of the experiment is shown in Table 1.

Table 1. Influence of basalt fiber waste on water separation of grouting slurries

% additive	Water separation, ml / in cylinders with different slopes, degrees							
	Basalt		Asbestos		Dunesand		Marblepowder	
	0	45	0	45	0	45	0	45
0	8	10	8	10	8	10	8	10
0.2	7	11	9	11	8	11	9	11
0.4	6	9	8	10	9	11	10	12
0.6	5	7	7	10	9	12	9	11
0.8	3	4	9	9	8	10	8	10
0.10	2	3	5	5	7	9	7	9

Experiments have shown that all used forming additives reduced the water separation of the grouting slurries. At the same time, for basalt fiber, with an increase in the amount of additives, the water separation of both horizontal and inclined cylinders has much decreased than asbestos, marble powder and dune sand. Apparently, this is due to the formation of a stronger structure in the cement slurry, which retains free water. It should be noted that all types of fillers are characterized by a weak manifestation of the effect at their concentrations of 6%, which is obvious with their insufficient amount to create a structure capable of retaining free water in solution. Another research was devoted to the study of the influence of waste basalt fiber on the strength of cement stone. Table 2 shows the results, measuring the strength of the cement stone at the age of 5 to 20 days at various concentrations of waste basalt fiber.

Table 2. Influence of waste basalt fiber on the strength of cement stone

Fiber content in solution	Compressive strength, MPa at age, days				Tensile strength, MPa at the age of a day				Flexural strength, MPa at the age of a day			
	5	10	15	20	5	10	15	20	5	10	15	20
0	23.8	35.8	39.0	47.1	0.77	0.98	1.37	1.53	6.09	7.77	8.90	8.75
2	28.7	34.1	44.3	55.6	0.95	1.24	1.49	1.71	6.70	7.93	9.17	9.47
4	29.5	39.0	47.5	57.4	1.33	1.42	1.57	1.86	7.10	8.30	9.00	9.72
6	30.6	43.3	49.6	58.7	1.46	1.53	1.61	1.95	7.41	8.52	9.25	9.98
8	31.9	45.7	56.6	60.0	1.57	1.60	1.79	2.15	7.65	8.80	9.53	10.33
10	33.4	49.6	58.5	65.4	1.66	1.78	1.89	2.37	7.91	8.97	9.89	10.57

Studies have established that at the age of 20 days, fibers at a concentration of up to 10 % significantly affect the strength in all types of destruction. At shorter curing times, the effect of fiber is most noticeable for the tensile strength of the cement stone, and to a lesser extent for the flexural and compressive strength.

In the next series of experiments was devoted to the study of the effect of waste basalt fiber on the permeability of cement stone. The research results are presented in table 3.

Table 3. Influence of basalt fiber waste on the permeability of cement stone (W/C=0.5)

Curingtime, perday	Permeability of mDarsi cement stone at different fiber concentration					
	0	2	4	6	8	10
5	195.3	158.3	127.4	103.7	114.1	65.6
10	10.8	8.8	6.9	4.8	3.5	2.0
15	4.9	6.5	7.3	3.7	2.4	1.5
20	3.0	2.5	2.2	1.8	1.5	1.0

As can be seen from the data in Table 3, the addition of basalt fiber waste reduces the permeability of the cement stone, starting from low concentrations, and the positive effect of fiber with an earlier period can be explained with the formation of closed pores, which are most actively formed after 15 days of hardening.

As we know, one of the most important technological indicators of the grouting composition is volumetric expansion. When cement slurry hardens, two competing processes take place. The first is shrinkage caused by contraction, the second is expansion associated with an increase in the volume of the formed cement gel compared to the volume of cement. As you know, the total effect of these processes, influencing in different directions the change in the volume of the hardening system, and will determine the shrinkage or expansion after the complete completion of the process of formation of cement stone [2].

Since the addition of basalt fiber reduces shrinkage, even in the absence of expanding additives in the grouting slurry, it is possible to obtain an increase in the volume of the cement stone during hardening. In the case of using expanding additives, the expansion should be higher, due to the crystallization pressure of the expanding additive and will be transferred to the framework formed by basalt fiber. Experimental verification has confirmed the above assumptions. Perhaps this explains the increased grip with bounding surfaces. The research results are presented in table 4.

Table 4. Volumetric expansion of cement stone with the addition of basalt fiber

Astringent	W/C	Volume expansion, % during hardening of cement mortar with the addition of basalt fiber %					
		0	2	4	6	8	10
Air hardening							
Portland cement	0.5	0.14	0.16	0.18	0.21	0.29	0.33
Water hardening							
Portland cement	0.5	-0.2	-0.06	0.02	0.06	0.07	0.08

Today, both in Uzbekistan and in many oil and gas producing countries of the world, hydrocarbon reserves of raw materials are concentrated in fields with a high content (from 12 to 20 %) of hydrogen sulfide. Hydrogen sulfide has a destructive effect on cement stone, creating a serious environmental threat, both on the surface and in the subsoil surrounding the well. Currently, the grouting materials used in such fields practically do not provide the formation of a reliable cement stone and its required durability in an aggressive environment. This is confirmed by the observed increase in the number of inter-layer hardened crossflows associated with the deterioration of the cement stone (45-60 % of the total well stock).

In this regard, the next stage of the study was devoted to the study of the sulfur-hydrogen resistance of cement stone obtained with the addition of basalt fiber from 5 to 20 % of the weight of dry cement. For comparison, samples of a cement girder were made with sand and marble powder added to the cement mortar. The water-cement ratio was 0.50-0.55, the spreadability along the cone of AzNII was 19-20 cm. the study of hydrogen sulfide exposure consisted of two

stages. At the first stage, thin sections and beams were created, made of hardened compounds, which were exposed to an aggressive environment. The study of the effect of an aggressive environment on hardening cement stone samples was carried out on a unit prepared according to the NACE TM0177-96 standard, the diagram of which is shown in Fig. one.

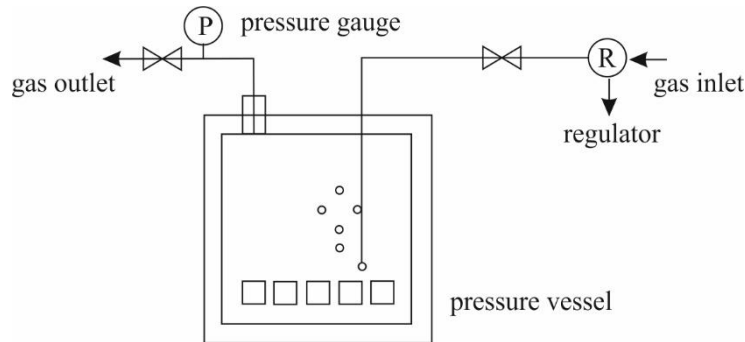


Figure: 1. Schematic diagram of the test setup

The mixing liquid temperature was maintained at 20 °C. The hardening medium of the cement stone samples was water, which was subsequently replaced by a corrosive mortar prepared according to the NACE TM0177-96 standard, and consisted of an acidic, H₂S-rich medium.

At the second stage, the samples were examined after 1 month, 6 months, 12 months and 36 months in order to study in detail the processes and reactions occurring with the cement stone during the specified period of time. The samples were stored in a desiccator maintained at a constant 100 % humidity. The main criteria for analyzing the structural state of a cement stone subject to hydrogen sulfide aggression were: porosity, strength, permeability, fracturing, as well as the structure of the formed cement stone. A JEOL JSM-6390A scanning electron microscope was used for research. The optimal percentage ratios of the reagents with the mixing water volume and with each other were selected empirically using the theory of experiment planning, considering their compatibility and the main technological parameters of the resulting grouting mixture and stone. The main parameters included fluid loss, spreadability, thickening time, setting time, flexural strength, sedimentation stability. The developed compositions of the grouting material, the mixture is presented in table 5.

Table 5. Developed plugging materials

Composition number	Composition of the grouting mixture
1	Portland cement (PC)+ fresh water (FW)+5% U-ROP
2	PC+FW+10.0% Basalt fiber (BF)+ 5% U-ROP
3	PC+FW +20.0% BF+5% U-ROP
4	PC+FW +10.0% Marble powder (MP)+5% U-ROP
5	PC+FW +20.0% MP+5% U-ROP
6	PC+FW +10.0% Dune sand (DS)+5% U-ROP
7	PC+FW +20.0% DS+5% U-ROP

The best indicators for the studied parameters were achieved for composition 3, compositions 4-7 are given as a comparison at percentage ratios, composition 1 acted as reference.

The available and inexpensive reagent U-ROP (Universal reagent for oil production), representing (20-45 %) an aqueous solution of sodium salts of mono- and dicarboxylic acids, which interact with calcium ions and other components of the cement slurry, was used as a plasticizer. with the formation of organo-mineral compounds that fill the pore space during the formation of the plugging stone, which prevents the penetration of hydrogen sulfide into the cement stone.

In our opinion, the resistance of the cement stone to the aggressive effects of hydrogen sulfide of compositions 2 and 3 is provided due to the chemical condensed micro-dispersed particles by nature different from the particles of Portland cement. Comparison of the main parameters of the listed cement slurries is shown in Fig. 2.

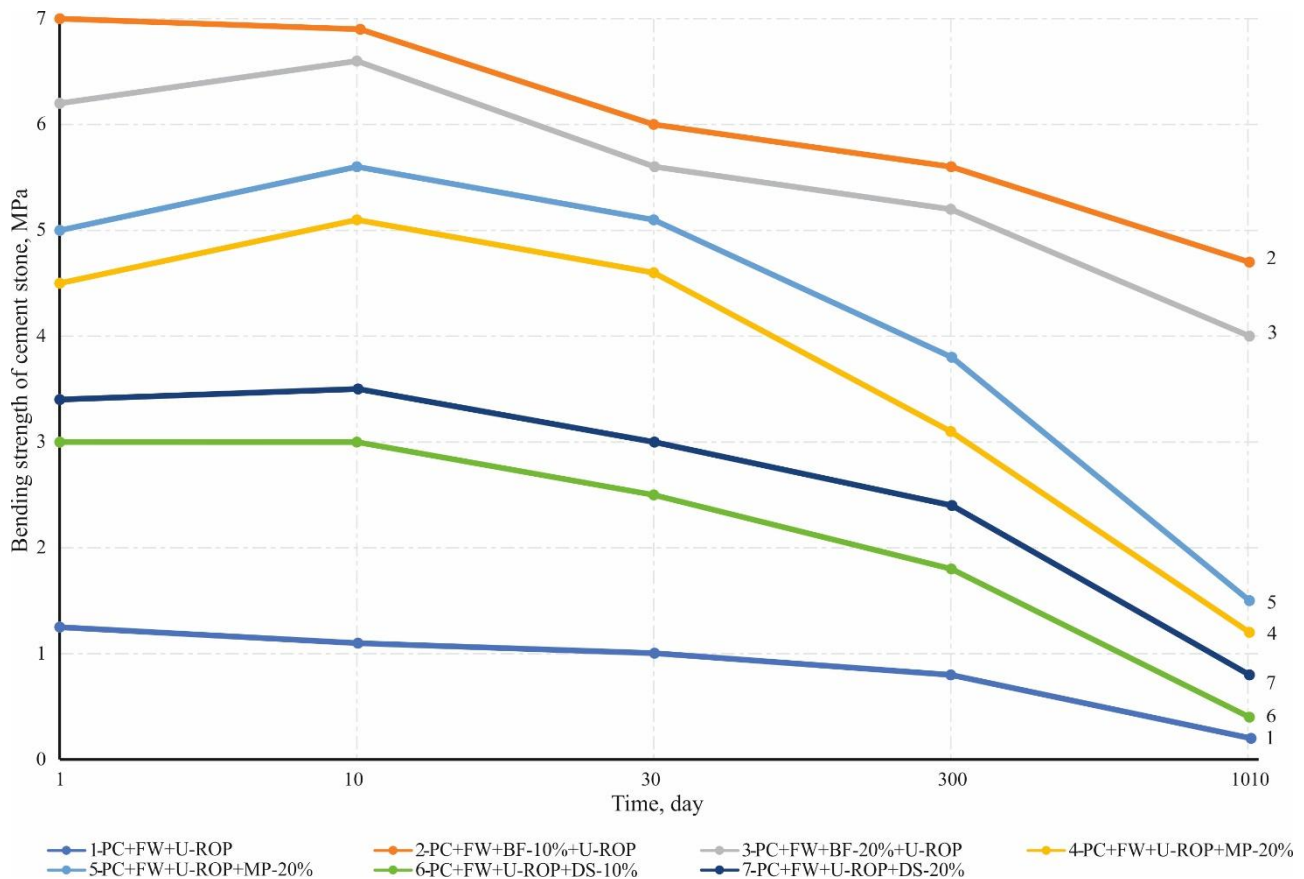


Figure: 2. A graph of the dependence of the change in the bending strength of a cement stone over time

Analyzing graph 2, it can be stated that the presence of corrosion-resistant materials does not impair the strength property, but can be improved by creating an impermeable structure. In general, cement stone obtained on the basis of Portland cement with the addition of 10 to 20 % waste of basalt fiber to provide resistance against the aggression of H₂S for a long time in comparison with marble powder and dune sand.

The next stage of the study was devoted to the study of the degree of strength to salt aggression of cement stone obtained with the addition of Portland cement from 5 to 20 % of basalt fiber waste. To compare the salt resistance of the stone, marble powder and sand dunes were studied, adding 20 % of each to the Portland cement of the Sherabad cement plant of Uzbekistan. The prepared standard size cement beam was left at 20-25 °C in a desiccator. It should be noted that the stability of cement stone in highly concentrated salt solutions has been poorly studied. Today, there are a number of methods for studying the resistance of cement stone to salt aggression. According to [3], the most simple and accurate is the study of cement stone to the aggressive effect of salts through the strength characteristics of the cement stone and the loss of their weight after salt contact. Therefore, we have studied the stability of cement stone in highly concentrated solutions of chlorides and sulfates. The results of the study are presented in table 6. The data in table 6 indicates a higher salt resistance of cement stone, which contains up to 20 % waste of basalt fiber. Marble powder and dune sand of the same dosage are far behind in terms of the studied indicators of basalt fiber.

The final stage of the study was devoted to the study of high temperature on the quality of cement stone.

The temperature factor has a huge impact on the reliability and durability of backfill cement and technology of deep well casing, as well as the process of oil production intensification due to thermal impact on the reservoir by steam injection, where the temperature in the well sometimes exceeds 300 °C [4].

In these conditions, it is necessary to use only heat-resistant grouting compositions. In all four oil and gas producing regions of Uzbekistan, hydrocarbon reserves are almost established, having drilled wells up to 4-4.5 km, where the reservoir temperature did not outweigh 100-10 °C. In the future, it is planned to drill deep and super-deep wells 7 km deep, where the temperature in the wellbore reaches 160-170 °C. In this regard, the development of a backfill stone resistant to temperature aggression is considered the most urgent.

In order to study the influence of the temperature factor, we prepared a grouting solution with the addition (each separately) of 10, 20 and 30 % (by weight) of waste of basalt fiber, blast furnace slag and natural basalt rock.

The grouting slurry had W/C=0.55 and spreadability along the AzNII-20 cone. The shape of the beams for studying the strength characteristics of the cement stone is standard. The maximum temperature regime was created using a muffle furnace down to -160 °C. The maximum contact time with temperature is 180 hours. The results of high temperature studies are presented in Table 7.

Table 7. Influence of high temperature on the main technological properties of cement stone formed with the presence of various Internet additives

№	Characteristics of cement stone	Additive name, content in% by mass								
		Waste basalt fiber			Blastfurnaceslag			Naturalbasaltrock		
		10	20	30	10	20	30	10	20	30
1	Compressive strength, MPa	64	75	34	45	43	66	57	68	76
2	Flexural strength, MPa	13	17	21	9	13	17	10	14	18
3	Thickening time of the cement slurry, h, min	1.00	1.30	2.0	1.10	1.30	1.45	0.50	1.20	1.50
4	Shrinkagein%	0.36	0.26	0.20	0.20	0.26	0.30	0.35	0.30	0.2

IV. CONCLUSION

The data in Table 7 indicates the resistance to elevated temperatures of cement stone containing up to 30% waste of basalt fiber. In terms of strength and other characteristics, both basalt rock and basalt fiber waste are much superior to blast furnace slag in terms of resistance to temperature aggression.

Thus, the addition of basalt fiber waste to the grouting mortar gives the cement stone the following valuable qualities:

- reduces the density of the solution from 1850 to 1410 kg/m³. No lightening additives;
- reduces water separation of the cement slurry;
- increases the compressive, bending and tensile strength of the cement stone;
- reduces permeability, increases the plasticity of cement stone;
- has a moderate expanding ability;
- creates a high resistance to corrosion of cement stone at hydrogen sulfide and salt aggression;
- increase the resistance of the cement stone to high temperature aggression.

A complex acting additive - waste of basalt fiber and reinforcement are recommended to be used for cementing wells in the section of which one or several types of aggressive formation fluids are contained.

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Composition of the grouting solution	Percentage of additive in cement slurry	Ultimate strength of cement stone at the age of 20 days before contact with salts, MPa			Weight loss in % of cement stone after 1010 days of contact with salts		Ultimate strength of cement stone after contact with salts, MPa			The quality of the contact of the cement stone with the rock casing
		Compression	Stretch	Bend	Chlorides (halite, sylvinitofite) 25 % each	Sulfates (gypsum, kieserite anhydrite) 25 % each	Compression	Stretch	Bend	
Portland cement	-	47.1	1.53	8.75	13.7	10.9	34.8	1.0	6.25	A gap of up to 2mm has formed
Basaltfiber	5	51.6	1.71	9.15	13.3	10.8	50.5	1.65	9.00	A gap of up to 1mm has formed
Basaltfiber	10	54.4	1.86	10.77	10.1	9.3	52.2	1.77	10.35	Weakcontact
Basaltfiber	15	57.5	1.95	12.51	8.5	7.0	56.3	1.81	12.10	Satisfactory
Basaltfiber	20	65.7	2.15	15.10	5.7	4.2	64.9	2.05	14.9	Qualitative
Marble powder	20	56.8	1.88	10.2	10.0	9.5	55.0	1.70	8.93	Weakcontact
Dune sand	20	52.3	1.71	9.17	11.6	10.7	48.7	1.57	8.00	Weakcontact

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