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Researches on the Use of Metakaolin to Improve the Properties of Pastes Used in Oil Well Cementing

Magdalena Banu

Muzeul de Ştiinţe Naturale al Judeţului Prahova, Str. Erou Călin Cătălin, 1, Ploieşti
e-mail: banumagdalena@yahoo.com

Abstract

The introduction of Pozzolanic materials in the cement, as Supplementary cementitious materials (SCM), has become during the last few years a successful worldwide practice. It has been proved that these can react chemically with the hydrated cement, forming a modified microstructure of the paste, leading to the improvement of the cements properties with respect to the compressive strength, the durability and the behaviour in aggressive environments or at high temperatures.
In this study we have analysed the effect of adding metakaolin, in 10%-20% proportion, on the properties of the pastes that may be used in cementing oil wells. Four products of the metakaolin category have been used for testing, under the trade name of Power Pozz™HRM , Metaver™ R, Metaver™ W and Microsit® M10, manufactured by Advanced Cement Technologies USA and NewChem Switzerland. The researches made in the laboratory have studied the influence of such admixtures on the set time, on the spreading, density, compressive strength and shrinkage of the pastes achieved with such mixtures.

Key words: *puzzolana, metakaolin, durability***,** *compressive strength, initial and final set time, shrinkage*

Introduction

The introduction of pozzolanic materials in the cement, as Supplementary cementitious materials (SCM), has become during the last few years a successful worldwide practice.

The Pozzolanic materials are „siliceous or siliceous and aluminous material, which in itself possesses little or no cementing property, but will in a finely divided form - and in the presence of moisture - chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties." [1]

They can be classified into two main groups – natural products (volcanic tufa, rocks of meteoric impact, diatomeic soils, bauxite) and artificial products (baked clays and schists, thermal power station ashes with low content of calcium oxide, silica fume collected from the gases exhausted during the production process in the metallic silicium and alloy plants etc). [2] [3]

The results of the researches made so far on the use of the pozzolanic materials obtained through clay baking as Supplementary cementitious materials (SCM), have encouraged us to test their applicability on achieving some improved compositions for oil well cementation.

The baking of clays at high temperatures leads to the formation of some metastable phases of transition which show a special pozzolanic reactivity both due to their great area and to their amorphous nature. A typical representative of this metastable phase, successfully used during the last years in the cements industry, is the product used through the baking of kaolin, named *metakaolin.* [4]

In this study we are presenting the results of researches made in the laboratory on the properties of the cement pastes in which 10%-20% metakaolin has been added. The tests made have analysed the influence of such admixtures on the set time, on the spreading, density, compressive strength and shrinkage of the pastes achieved with such mixtures.

The Materials Used in the Tests

Metakaolin

In order to prepare the pastes 4 types of metakaolin have been used, under the trade name of Power Pozz™HRM , Metaver™ R, Metaver™ W and Microsit® M10, supplied to us by Advanced Cement Technologies USA and the company NewChem AG Switzerland through Meselia Expert Solutions s.r.l., its dealer for Hungary and Romania.

According to the data sheets of the products [5-8] and the ASTM C618 requirements on Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete (Class N), these 4 types of metakaolin show the following physico-chemical properties, presented in Tables 1 and 2:

Table 1. Physical properties of metakaolins [5-8].

Pozzolan Name	**Specific Density** g/cm^3	**Apparent density freely settled** g/cm^3	**Apparent Density tapped** g/cm^3	**Colour**	**Specific surface Blaine** cm^2/g	**Specific surface BET** m^2/g	**Distribution of particle size**
Power Pozz™ HRM	2.6	0.4		whitish		15	D10 / D50 / D90 2µm/4,5 µm/14 µm
Metaver™ W	2.6	0.4-0.5	cca 0.6	cream	12,000	15	D10 / D50 / D90 2µm/10 µm/25 µm
Metaver™ R	2.6	0.5-0.6	cca 0.9	red	10,000	17	D10 / D50 / D90 2µm/30 µm/100 µm
Microsit ® M10	2.49	0.66	0.87	grey			D10 / D50 / D95 ≤2µm/≤4 µm/≤10 µm

Table 2. Chemical properties of metakaolins [5-8]

Pozzolan Name	**SiO2**	**Al2O3**	**Fe2O3**	**TiO2**	**SO_3**	**CaO**	**MgO**	**Na2O**	**K2O**	**Lost at ignition**
Power Pozz™ HRM	52-54	42-44	<1-1.4	<3.0	<0.1	<0.1	<0.1	<0.05	<0.4	<1.0
Metaver™ W	54-56	36-38	<1.9	<1.0	<0.8	<0.9	<0.6	<0.4	<2.8	<2.5
Metaver™ R	67-69	25-27	<2.5	<1.5		<0.8	<0.1	<0.1	<0.2	<1.5
Microsit ® M10	52	26	6			5				3,5

Power Pozz™HRM is a whitish, amorphous product, obtained through the thermal treatment of purified kaolin which strongly and rapidly reacts with the calcium hydroxide forming compounds with cementing properties. [9]

The material is 99.9% finer than 16μm, and has a mean particle size of 3μm (as measured by MicroTrac laser diffraction granulometer method). The PowerPozz[TM] HRM Particle Size Distribution is illustrated below in Figure 1 [5] :

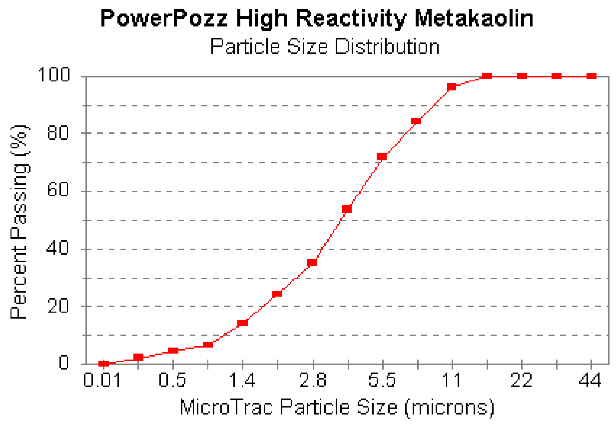

Fig. 1. Particle Size Distribution of PowerPozz[TM] HRM[5]

Metaver[TM] R is a reddish material, produced by calcination of kaolin, mostly amorphous alumosilicate, considered to be very reactive because together with lime and water it starts hardening after about 3 hours. It may be used to replace the cement in a 5-20% proportion in weight. [6]

Metaver[TM] W is produced by calcination of concentrated kaolin and is a whitish, mostly amorphous alumosilicate, considered to be moderately reactive because together with lime and water it starts hardening after about 22 hours. It is indicated to replace the cement in a 5-15% proportion in weight. [7]

Microsit® M10 is a new additive for the controlled production of high-quality mortars and concretes. The product is grey and consists mostly of SiO_2 and Al_2O_3 and is classified as an aluminosilicate. Microsit® M10 reduces the amount of water required to make the pastes and improves their rheological properties. The optimization of the field of fine particles in the mixture leads to obtaining an increased density and durability of the links in the cement matrix. It may be used to replace the cement in an 8-15% proportion in weight, function of the desired properties [8].

Table 3. Physical and chemical properties of the Portland Cement CEM I 32,5N-LH [10].

	Characteristics								
	Chemical				**Physical**			**Mechanical**	
	PC (%)	**Residue insoluble in HCl (%)**	**SO_3 (%)**	**Cl^- (%)**	**Hydration heat (J/g)**	**Initial set time (min)**	**Stability (mm)**	**7 days (MPa)**	**28 days (MPa)**
Lot tests results	0.37	0.15	2.75	0.0103	259	185	1,0	36.8	47.8
Standard requirement	max 5	max. 5	max 3	max. 0.10	max. 270	min. 90	max. 10	min. 16	32.5-52.5
Testing method	SR EN 196-2				SR EN 196-8	SR EN 196-3		SR EN 196-1	

Portland Cement

In order to make the tests it has been used a Portland Cement CEM I 32.5N-LH, with reduced hydration heat, produced by SC LAFARGE CEMENT (Romania) S.A., in its Medgidia plant, in the lot No.1264.

According to the laboratory test report, this cement, with a standard content of 95-100% Portland clinker and a maximum amount of 3.5% C_3A, has the chemical, physical and mechanical properties, presented in Table 3. [10]

Preparation of the Pastes

Mixtures of cementing powder and metakaolin have been made, in a proportion of 10%, 15% and 20% of the mixture weight, using each of the testing products Power Pozz^{TM}HRM, Metaver^{TM} R, Metaver^{TM} W and Microsit$^{®}$ M10. In order to get pastes as homogeneous as possible, the solid components were blended in the mixer for 5 minutes, then water was added and it was mixed on for 5 more minutes. The pastes were made using a water/mixture ratio of 0.5.

Methods Used and Test Results

The Initial and Final Set Time

Time to initial and final set was measured using a Vicat apparatus. Three samples were used for each measurement at 20±2^{0}C. The preparation ratio of the pastes used was W/C = 0,5.

Tests have been made on a control sample of simple cement (C) and on 12 compositions with replacement metakaolin admixture in 3 successive concentrations -10%, 15%, 20%, of each studied product - Power Pozz^{TM}HRM, Metaver^{TM} R, Metaver^{TM} W and Microsit$^{®}$ M10. The results obtained are presented in Table 4.

Table 4. The initial and final set time of the pastes.

Sample code	Composition of the paste	Initial set time	Final set time
C	SIMPLE CEMENT	5h15”	6h50”
PP1	C+POWER POZZ 10%	4h55”	6h25”
PP2	C+POWER POZZ 15%	4h30”	6h15”
PP3	C+POWER POZZ 20%	4h	5h
MR4	C+METAVER R 10%	5h30”	6h45”
MR5	C+METAVER R 15%	5h45”	7h
MR6	C+METAVER R 20%	6h	7h15”
MW7	C+METAVER W 10%	3h25”	5h40”
MW8	C+METAVER W 15%	3h45”	6h45”
MW9	C+METAVER W 20%	5h	7h15”
MICR10	C+MICROSIT 10 10%	4h45”	9h35”
MICR11	C+MICROSIT 10 15%	7h30”	10h45”
MICR12	C+MICROSIT 10 20%	12h45”	15h15”

The laboratory tests have underlined the fact that the substitution in moderate amounts of the cement with Power Pozz^{TM}HRM (10-20%) in the cement pastes leads to a reduction of initial set time (by 20 minutes - 1h 15 minutes) and final set time (by 25 minutes – 1h 50 minutes), as compared to the control samples of simple cement, made under the same conditions. The reduction of the set time is pro rata with the amount of cement replaced with metakaolinic admixture. These results can be due to the high proportion of Al_2O_3 (42-44%) contained by Power Pozz^{TM}HRM and its very reactive character as compared to the calcium hydroxide produced in the cement hydration process, by which it forms stable products.

The tests made with Metaver^{TM} R have shown values comparable to those of the cement, at a low replacement of 10% and slightly higher (by 15-45 minutes of the initial time and by 10-25 minutes of the final time) for those with 15% and 20% metakaolin. In these samples the increase of the set time is pro rata with the amount of cement dislocated. This behaviour may be induced by the higher amount of SiO_2 (67-69%) contained by Metaver^{TM} R.

The samples prepared with Metaver^{TM} W have shown a significant reduction of the set times for the replacement percentage of 10% and 15%, higher than that of the pastes prepared in the same proportions with Power Pozz^{TM}HRM. Such behaviour may be induced by the higher amount of SiO_2 (54-56%) and the presence of higher percentage of basic oxides (CaO and MgO) and acid oxides (SO_3) contained by Metaver^{TM} W, as compared to Power Pozz^{TM}HRM
The greatest initial time reduction (by 1h 50 minutes) and final time reduction (by 1h10 minutes) have been noticed in the samples with 10% Metaver^{TM} W. The samples with 15% metakaolin have had a reduction of the initial set time by 1h 30 minutes, but the final set time is comparable to the one of the cement in the control samples. The tests with 20% Metaver^{TM} W have had values comparable to the ones of the simple cement, with variations of ±15 minutes.

The samples with Microsit® M10 have generally had much longer set times. The exception is the sample with 10% substitute which has had an initial set time by 30 minutes shorter than the control sample, but a final time by 2h 45 minutes longer. The samples with 15% Microsit® M10 have had a growth of the initial set time by 2h 15 minutes and of the final time by 3h 55 minutes. The pastes with 20% substitute have shown especially long set times (over 12h), which cannot recommend them for practical applications. Actually the producer indicates the use of Microsit® M10 to replace the cement in a proportion of only 8-15% in weight. [8].

Such behaviour can be attributed to the characteristics of this metakaolinic admixture, the specific distribution of the size of its particles and their spherical form, which induce a growth of the flowing properties of its mixtures with the cement.

The Compressive Strength

The compressive strength has been determined by help of a hydraulic press, on cylindrical samples with diameter and height of 30 mm, at a temperature of 20 ± 2^0C. The preparation ratio of the pastes used has been W/C = 0.5. Tests have been made on a control sample of simple cement (C) and on 12 compositions of cement with metakaolin admixture in successive concentrations -10%, 15%, 20%, of each studied product - Power Pozz^{TM}HRM, Metaver^{TM} R, Metaver^{TM} W and Microsit® M10. The results obtained, measured in daN/cm^2, are presented in Table 5.

It has been noticed that the pastes in which the cement was replaced with Power Pozz^{TM}HRM have compressive strength 2-2.5 times higher than the control sample during the first 24 hours and maintain their superiority at all other time intervals. The remarkable values are those from 28 days to 90 days which are much higher than those of the cement.

Ranging second as performance, the pastes with Metaver^{TM} W have shown compressive strength 1.5-2 times higher than the control sample, during the first day. During the following time intervals (3-28 days), only the samples with 10% metakaolin have similar behaviours with the cement, the others showing lower values. Yet at 90 days, irrespective of the concentration, all the mixtures with Metaver^{TM} W have higher strength as compared to the control samples.

Table 5. Compressive strength of the pastes at 20^{0}C.

Composition of the paste	**Compressive strength (daN/cm^2)**				
	1 day	**3days**	**7days**	**28days**	**90days**
SIMPLE CEMENT	24.32	61.20	65.40	83.85	120.74
C+POWER POZZ 10%	76.30	79.65	88.88	142.54	187.81
C+POWER POZZ 15%	72.95	76.30	85.52	135.82	171.04
C+POWER POZZ 20%	69.59	70.43	83.85	130.80	153.44
C+METAVER R 10%	31.02	36.05	45.28	62.05	149.24
C+METAVER R 15%	25.99	29.35	39.40	53.67	139.18
C+METAVER R 20%	20.96	23.48	31.86	45.28	129.96
C+METAVER W 10%	56.18	61.21	62.88	77.98	135.83
C+METAVER W 15%	46.95	55.38	57.85	69.59	126.60
C+METAVER W 20%	38.57	41.08	46.11	58.69	122.41
C+MICROSIT 10 10%	24.32	31.86	34.38	63.72	121.58
C+MICROSIT 10 15%	21.80	27.67	31.02	57.01	112.35
C+MICROSIT 10 20%	19.28	25.15	28.51	48.60	103.13

The samples with Metaver^{TM} R 10% have shown a little higher values and those with 15% have had values comparable to the control ones, during the first day. During the following time intervals (3-28 days), they have had much lower strengths than the cement, but at 90 days all mixtures with Metaver^{TM} R have shown higher values than the control samples. It must be also noted that at 90 days, the samples with Metaver^{TM} R have shown superior strengths as compared to the samples with Metaver^{TM} W.

As far as the pastes prepared with Microsit® M10 are concerned it is noticed that only those where the replacement is 10% are comparable to the control samples at one day and at 90 days. The other concentrations have shown lower values of the strength for the entire duration of the test.

It must be noted that the data obtained after measuring the compressive strength during the first 24 hours are in compliance with the results noticed in the testing of the set time for the 4 types of compositions.

Table 6. Compressive strength, density and spreading of the pastes with 20% metakaolinic substitute

Composition of the paste	**Compressive strength after 1 day** (daN/cm^2)		**The density of the paste** (kg/dm^3)	**Spreading** (mm)
	at 20^{0}C	**at 60^{0}C**		
SIMPLE CEMENT	24.32	74	1.83	220
C+POWER POZZ 20%	69.59	94	1.80	185
C+METAVER R 20%	20.96	78	1.82	215
C+METAVER W 20%	38.57	80	1.78	190
C+MICROSIT 10 20%	19.28	86	1.8	230

In parallel with these determinations, tests have been made on the compressive strength at 60^0C, after 24 hours, on the samples with 20% metakaolinic admixture, using all the 4 products and an W/C ratio of 0.5. It has been noticed that the values obtained in the samples hardened at 60^0C temperature are much higher, almost double, for all the analysed mixtures. This encourages us to believe that the pastes with metakaolinic admixture can have a proper behaviour in the depth probes. The results of the tests are presented in Table 6.

The Spreading and Density of the Pastes

In parallel with studying the compressive strength, determinations have been made on the density and the spreading of the pastes with the highest content of metakaolinic substitute (20%). The results obtained are presented in Table 6.

It has been noticed that the densities of the pastes with metakaolinic mixtures are comparable with those of the cement, but with slightly lower values, by 0.3-0.5 units.

The pastes with Power Pozz™HRM and Metaver™ W have a smaller spreading than those made of simple cement, and those with Microsit® M10 exceed the value of the control samples.

Chemical Shrinkage

According to the Japanese Institute of Concrete [11], the chemical shrinkage (Le Chatelier) represents the variation of volume between the absolute volume of the hydration products on the one hand and the volume of water and of the cement on the other hand:

$$V(\text{hydrated constituents}) < V(\text{anhydrous constituents}) + V(\text{water}) \qquad (1)$$

According to the studies of several authors, the chemical shrinkage corresponds to a reduction of the absolute volume by 7-10% as compared to the initial paste volume. [12]

For the analysis of the chemical shrinkage of the mixtures cement -metakaolin we have used the dilatometric method, adapting the procedure described by the researcher Bresson A. [12] The tests have been made using pastes prepared under the same conditions of concentration, W/C ratio and temperature with the ones used to determine the parameters previously analysed.

Fig. 4. Measuring the chemical shrinkage through the dilatometric method.

In order to notice the chemical shrinkage a system has been used formed of a glass vessel with tight cap where a graduated pipette is introduced. Inside the vessel a paste volume of 100 ml is introduced, freshly prepared, then the recipient is filled with distilled water and the cap is fixed. Through the pipette, by help of a syringe, a surplus of water is introduced until reaching the 0 ml reference point, then a paraffin oil drop has been added to restrain the evaporation of water during the determination. The whole system has been introduced in a thermostatic precinct at 20^{0}C, and the readings have been made through the protection window, for the exterior temperature not to influence the variation of the water volume. Accepting the hypothesis that the water fills under these conditions all the holes, the decrease of the water volume in the pipette corresponded to the change of the absolute volume of the paste sample, which is the chemical shrinkage.

The chemical shrinkage was expressed in percentage as compared to the initial volume of the paste. The results obtained are presented in Table 7

Table 7. Chemical shrinkage of the pastes (%)

Composition of the paste	**Variation of volume - chemical shrinkage Cs%**									
	3h	**6h**	**9h**	**1 day**	**2 days**	**3 days**	**7 days**	**14 days**	**28 days**	**90 days**
SIMPLE CEMENT	+0,05	+0,10	-0,2	-1,91	-2,57	-3,07	-3,72	-3,72	-3,82	-4,82
C+POWER POZZ 10%	0	-0,20	-0,41	-2,26	-2,58	-3.09	-4,23	-4,23	-4,43	-5,36
C+POWER POZZ 15%	0	-0,45	-0,75	-2,20	-3,10	-3,45	-4,30	-4,30	-4,60	-5,35
C+POWER POZZ 20%	0	-0,59	-0,92	-2,16	-3,07	-3,42	-4,46	-4,52	-4,85	-5,45
C+METAVER R 10%	+0,20	+0,05	-0,10	-1,40	-3,00	-3,30	-4,40	-4,40	-4,70	-5,30
C+METAVER R 15%	+0,25	+0,15	-0.10	-1,20	-2,20	-2,80	-3,60	-4,18	-4,47	-5,40
C+METAVER R 20%	+0,20	+0,05	-0,14	-1,30	-2,33	-2,54	-3,50	-4,35	-4,50	-5,50
C+METAVER W 10%	+0,20	+0,12	-0,10	-1,10	-2,52	-3.13	-3,94	-4,24	-4,44	-4,55
C+METAVER W 15%	0	+0,10	-0,10	-1,60	-1,70	-2,80	-3,60	-4,00	-4,30	-4,50
C+METAVER W 20%	0	0	-0,20	-1,40	-2,50	-2,85	-3,60	-4,00	-4,20	-4,40
C+MICROSIT 10 10%	+0,28	+0,40	+0,4	-1,40	-2,10	-2,60	-3,20	-3,40	-3,70	-4,70
C+MICROSIT 10 15%	+0,18	+0,30	+0,31	-1,33	-2,24	-2,50	-3,27	-3,32	-3,78	-4,90
C+MICROSIT 10 20%	+0,15	+0,21	+0,52	-1,35	-1,48	-2,45	-3,07	-3,44	-3,85	-5,42

The tests have stressed the fact that after 1 day, the samples with Power Pozz^{TM}HRM show a shrinkage greater than of the pastes with simple cement, but all the samples with Metaver^{TM} R, Metaver^{TM} W and Microsit$^{®}$ M10 have shrinked less than the control pastes.

After 7 days the average shrinkage of the cement samples is 3.72%. The pastes containing mixtures with Power Pozz™HRM in all the 3 concentrations, after the same interval, show shrinkage comprised between 4.23-4.46%, and the mixtures with 10% Metaver™ R, a similar value of 4.40%. All the other mixtures show smaller shrinkage than those of the simple cement. After 28 days the average shrinkage of the cement samples is 3.82%. Except for the pastes made with Microsit® M10, showing smaller values or comparable to the cement, all the other mixtures show a little higher values than the control pastes comprised between 4.20-4.85%.

After an interval of 3 months, the shrinkage of the control samples reaches 4.82%. The pastes with mixtures of Power Pozz™HRM and Metaver™ R have a little higher shrinkage than the simple cement, comprised between 5.30-5.50%. Unlike them, the samples with mixtures of Metaver™ W show a little lower values than the cement (4.40-4.50%), and the ones made with 10% and 15% Microsit® M10, values of 4.70-4.90%.

All these observations show that, in general, the replacement of the cement with metakaolinic admixtures leads to obtaining some pastes with better characteristics or at least comparable to those of some pastes made of simple cement which recommends them for cementing oil wells.

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Cercetări privind utilizarea metacaolinului pentru îmbunătățirea proprietăților pastelor folosite la cimentarea sondelor

Rezumat

Introducerea produselor de natură puzzolanică, ca materiale de înlocuire (SCM) în ciment, a devenit, în ultimii ani, o practică de succes pe plan mondial. S-a dovedit că acestea reacționează chimic cu cimentul hidratat formând o microstructură modificată a pastei care conduce la îmbunătățirea proprietăților lor în ceea ce priveşte rezistența la compresiune, durabilitatea şi comportarea în medii agresive sau la temperaturi ridicate.

În cadrul acestui studiu am analizat efectul adăugării metacaolinului, în proporție de 10%-20%, asupra proprietățile pastelor de ciment care pot fi folosite la cimentarea sondelor. Pentru testare s-au folosit patru produse din categoria metacaolinului, denumite comercial Power Pozz™HRM , Metaver™ R, Metaver™ W şi Microsit® M10, fabricate de către Advanced Cement Technologies USA şi NewChem Elveția . Cercetările realizate în laborator au urmărit influența acestor adaosuri asupra timpului de priză, răspândirii, densității, rezistenței la compresiune şi a contracției pastelor realizate cu astfel de mixturi.