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WASTEWATER SEPARATION FROM GYPSUM SUSPENSIONS AND THE MANAGEMENT OF RESULTING WASTE

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Abstract

In this work, the examination of separation of gypsum from wastewater generated during the machining of casts and the method of waste gypsum management have been presented. This waste gypsum is produced in Ortolab Poland facilities. Gypsum casts are cut and ground in water-cooled circular saws. Wastewater, with over-normative content of suspension, 10000 mg L^{-1} and sulphates, 1600 mg L^{-1} is produced in the process. The analysis of water and wastewater management in Ortolab Poland facilities led to propose the technique which included the construction of sedimentation tanks and the manner of gypsum separation by means of a flocculant. Obtained waste gypsum with water content of approximately 43% was solidified by two methods. In the first method the waste gypsum was solidified with process water by adding Portland cement, building plaster and activators ($MgSO_4$, NaCl, $Al_2(SO_4)_3$). In the second method the waste gypsum was filtered anddehydrated. Setting time, water/gypsum ratio and compressive strength of solidified samples have been determined for both methods. The structure of materials have been determined by SEM while the phase was analyzed using XRD method. Gypsum content in materials have been determined by TG/DTA method. The strength of the solidified gypsum mixture after dehydration is 8.5 MPa. The strength of the solidified gypsum mixture without the dehydration process has uniaxial compressive strength equal to approximately 2.8 MPa and it cannot be a substitute of building plaster. The proposed dissolution of waste gypsum solidification at the site of its formation solves the problem regarding slurry transportation. It is an energy-saving technique due to the lack of gypsum dehydration process and ecological, because it limits the quantity of sulphate ions SO_4^{-2} entered into the sewerage system.

Keywords: gypsum suspension, waste gypsum management, wastewater treatment

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1. Introduction

In forming techniques and treatment of gypsum elements, grinding and additional cutting to size of details is a common activity. Circular saws used in these processes are water-cooled. In this way dusting is avoided, however, it causes the formation of hydrated gypsum suspension. This wastewater is characterized by over-normative content of sulphate ions SO_4^{-2}, approximately 1600 mg L^{-1}, and suspension, 10000 mg L^{-1}. The European directives were implemented, with the aim of regulating the degree of pollution for water sources (Enea et al., 2017).The World Health Organization (WHO, 2004) has established the maximum tolerable level of sulphate ions in water as 500 mg L^{-1}, but many countries have recommended lower values, such as the 250 mg L^{-1} recommended by Brazil and the USA (EPA, 1991; Silva et al., 2010).

In large monitored enterprises, a ban on the disposal of sulphate waste solids and mineral suspensions (Jablonska, 2010) into local waters has been imposed. Small enterprises pose more difficulties in terms of localization and control due to their vast quantity and dispersion. They employ low effective technical solutions while disposing a considerable amount of wastewater into the sewerage system. It causes siltation and the necessity to clean the sewerage

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system. Ortolab Poland was one of these enterprises, which used simple serial design for sedimentation tanks (Fig. 1).

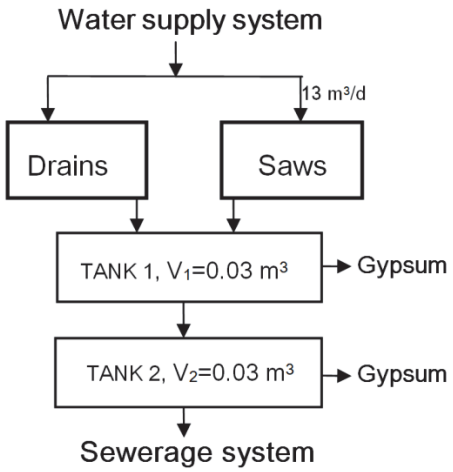


Fig. 1. The block scheme of the gypsum separation from wastewater generated during machining of casts in Ortolab Poland facilities

This installation does not meet the requirements pertaining to the allowed concentrations of suspension and SO_4^{2-} ions entered into the sewerage system. Technological assumptions covered the formulation of sedimentation tanks construction documentation, gypsum sedimentations with a flocculant and water circuit closure (Siedlecka at al., 2012). In the proposed technique gypsum is formed, and periodically removed from the sedimentation tanks. The removed gypsum contains 43% of water and small amounts of flocculant. Mixtures with the addition of building plaster, cement, and activators have been blended with hydrated gypsum and properties of the mixtures have been studied. Obtained results were compared with dehydrated gypsum and calcined at the temperature of 160 °C. Implementation of hydrated gypsum was to bind water containing sulphate within the mass of the formed material. Saving energy consumed during gypsum calcination is also important. Implementation of other substances into gypsum leads to changes in structures of strengths of obtained materials. The implementation of a flocculant into the process of separation gypsum results in the coverage of gypsum particles by the said agent. In addition, implementation of building plaster, Portland cement, and activators required basic examination to be conducted with reference to the received mixtures. The issue pertaining to modification of gypsum may be currently found in the works of Colak (2002), who seeks optimal relations between gypsum-cement-pozzolana, Hernández-Olivares et al. (1999), who describe techniques and properties of gypsum with cork fillers, Colak (2000) – examination of density and strengths of foamed gypsums. The influence of superplasticizers on temperature and setting time of agents: gypsum-cement-silica fume has been analysed by Kovler (1998). Laboratory and semi-technical tests, confirming the beneficial influence on the increase of strength values and acceleration of the setting process, pertaining to the addition of Portland cement and K_2SO_4 have been described by Singh and Garg (1995) and Singh (2005). The inclusion of waste gypsum to recycling process has been analyzed by Godinho-Castro et al. (2012). The influence of additions (sodium citrate, organic salts and acids, inorganic salts) on the microstructure and strength value of the bound gypsum is presented in works of (Combe and Smith, 1964; Koslowski and Ludwig, 1984; Lioliou et al., 2006; Murat and Jeandot, 1973). The influence of flocculants on flotation and flocculation properties of minerals and the changes of zeta-potential due to the use of flocculants have been analyzed by Salopek et al. (1992). The examination shows that zeta-potential of minerals with an addition of anionic flocculants (with high molecular mass weight) is negative. The influence of polymer flocculants depend much more on their molecular mass than ionogenity. The zeta-potential can help in the choice of flocculants but evaluation of the effectiveness can be given only after sedimentation or other suitable tests. A review of literature indicates that many factors, mainly, including chemical and crystallographic composition, pollutants, disintegration, implementation of an admixture, activators or retardants, influence the hydration process and the strength value of solidified materials. It unequivocally points to the necessity for carrying out laboratory tests on mixtures, which are to serve as materials used in the building industry. It concerns, to a large extent, materials composed of waste gypsums.

2. Material and methods

2.1. Wastewater from the techniques of gypsum model treatment before technology implementation

Gypsum used in casting techniques of gypsum models comes from the firm Zhermack-Italy. It is characterized by its high whiteness, water/gypsum ratio (w/g) is 0.3. Setting time is between 10-14 minutes, maximal linear expansion is 0.15%, and compressive strength of 25±2 MPa.

Wastewater with over-normative content of gypsum suspension, 10000 mg L^{-1} and SO_4^{2-} ions, approximately 1600 mg L^{-1} is formed during the treatment of gypsum models in the plant. In wastewater samples taken in the plant settleable solids have been tagged by volumetric and gravimetric method with respect to PN 72/C-04559.02, standard, SO_4^{2-} concentration of ions with respect to PN-EN 12457-4-2006. Gypsum content in raw material have been determined by Labsys derivatograph (TG/DTA). The TG/DTA curves were recorded in air atmosphere with a heating rate 10 °C min^{-1} to the final temperature 1000 °C. The sample mass was about

30 mg. Waste structure have been determined by electron microscope SEM (model JEOL JSM-6610LV) while the phase was analyzed using D8 Advance X-ray diffractometer (XRD).

2.2. Waste gypsum preparation for flocculant-supported sedimentation

Gypsum models have been crushed in the laboratory jaw crusher (type: LAB-01-65, EKO-LAB), and ground in the vibratory laboratory grinder (type: LMW, TESTCHEM). Gypsum has been acquired, which entirely went through the sieve holes when sifted through the sieve 63 μm, like the gypsum from the treatment of models. The examination of the sedimentation process covered: indication of the suspension by means of the gravimetric method with respect to PN 72/C-04559.02 standard, indication of the sedimentation time and the sediment volume of raw and flocculated suspension in accordance with PN 72/C-04559.03 standard, indication of the flocculants dose. Samples were analyzed by Labsys derivatograph (TG/DTA), X-ray diffractometer (XRD), and scanning electron microscope (SEM).

2.3. Waste gypsum solidification after technology implementation

Waste gypsum solidification was conducted in two ways. In the first method the waste gypsum was solidified without the dehydration process. In the second method the waste gypsum was filtered and dehydrated. The dehydration process was carried out at a temperature of 160 ^{o}C over a period of 3 hours. During the examination of solidification in the first method, particular binders were used: building plaster which meets the requirements of PN-EN13279-1:2000, Portland cement corresponding to PN 90/B 30010 TÜVCERT standard, analytically pure setting time activators (NaCl, $MgSO_4$ i Al_2SO_4) produced by the firm POCH Gliwice. In liquid mixtures the water/gypsum ratio (w/g) was calculated by the Shouthard's viscometer, while setting time was determined via the Vicat apparatus. Compressive strength was determined in the solidified samples by TONI NORM press, pressure: 100 kN, in accordance with the PN-EN-13279-1:2005 standard. Electron microscope (SEM), Labsys derivatograph (TG/DTA) and X-ray diffractometer (XRD) have been used in order to carry out the examination of the gypsum structure. The leaching of gypsum material (after dehydration at 160 °C) using water was carried out. Obtained results were compared with additional requirements for gypsum materials laid down by *Eurogypsum* (according to VGB instruction sheet VGB-M 701e, 2008).

3. Results and discussion

Wastewater examination after grinding gypsum models indicated that the content of gypsum in wastewater varies between 9.5 g L^{-1} to 10.1 g L^{-1}. Test average was 9.8 g L^{-1}. Sulphate concentrations were between 1440 to 1700 mg L^{-1}, average 1580 mg L^{-1}. The analysis of received findings indicates that SO_4^{2-} ion concentration is exceeded by more than three-times. Total suspended solids are exceeded 300 times. These excesses result in environmental fee charges. Sewerage system is often silted and needs cleaning. Existing system, which is employed to separate gypsum, envisages 2 cascade sedimentation tanks connected to form a row. In this technique adjunctive substances facilitating the gypsum separation process are not used (Fig. 1). Lack of efficiency pertaining to gypsum separation by sedimentation tanks used hitherto forced the implementation of a new method. It is based on the application of horizontal chamber sedimentation tanks. The sedimentation process is conducted with the usage of a flocculant. The study determined the type of flocculant, its concentration, process time, increase the volume of gypsum sediment in function of time (Fig. 2). There was also determined the suspension content in sludge supernatant (Fig. 3).

Sediment volume grows together with the flocculant concentration growth (Fig. 2). Sedimentation time decreases with the growth of flocculant concentration. The growth of flocculant concentration correspondingly decreases the content of gypsum in sludge supernatant reaching 7 g L^{-1} in sedimentation without the flocculant and 2.5 g L^{-1} when flocculant concentration is at the highest level (Figs. 2-3).

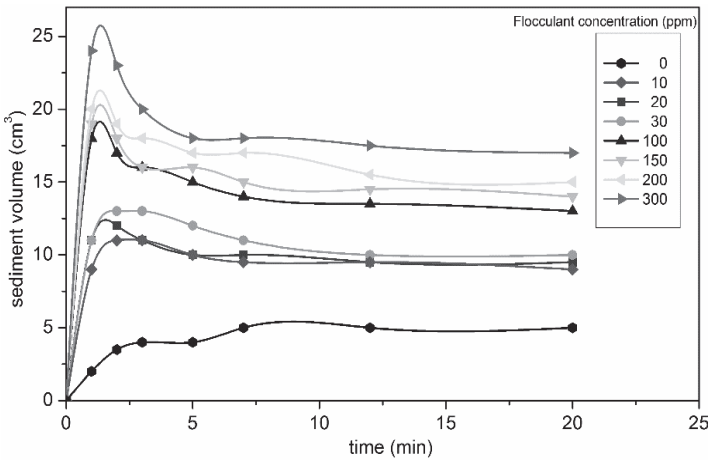


Fig. 2. The gypsum sediment volume dependence on flocculation time and flocculant concentration (0-300ppm)

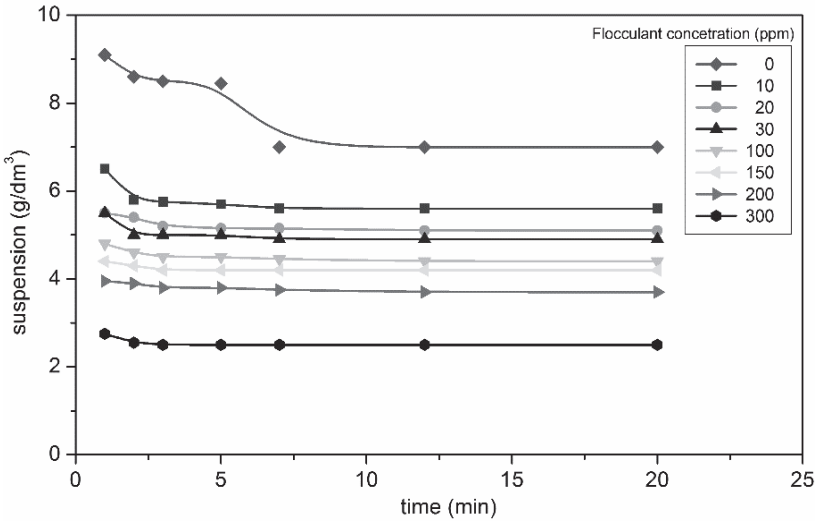


Fig. 3. Suspension content in sludge supernatant depending on flocculation time and flocculant concentration (0-300ppm)

For every dose of the flocculant, curves of suspension content, depending on the time, already reach a constant value after 3 minutes of the sedimentation process. Low content of suspension after the sedimentation process allows the water circuit closure and sludge supernatant usage in the grinding process.

Based on examination results and calculations made with respect to horizontal sedimentation tanks a new solution was proposed (Fig. 4). The addition of flocculant to the wastewater getting into tanks 1 and 2 caused separation of gypsum. Tank 3 is used to close water circuit through redrawing the treated liquid in order to reuse it in the cutting process and gypsum model grinding. Tank 4 functions as a buffer protecting the whole system from overfilling. Based on calculations, a technological system separating gypsum from wastewater was constructed and fitted in the plant (Fig. 5).

Gypsum sludge resulting from the system forms waste, which must be managed. In accordance with Commission Directive 2003/22/EC waste containing gypsum should be stored at separately isolated and properly secured landfills. It was noted, that gypsum stored together with municipal waste causes danger for natural environment. Processes pertaining to interactions between $CaSO_4$ and bacteria from municipal waste are the sources of the danger.

These processes generate hydrogen sulphide and other toxic substances, which may pass into groundwater, surface water and air. In accordance with WRAP Union programme (WRAP Plasterboard Programme, 2008), recycling is the fundamental course of economic exploitation.

The abovementioned findings favour the search for possibilities concerning management of gypsum from the system in question. The TG/DTA curves of waste gypsum before and after the process of flocculation (Fig. 6) indicated, that calcium sulphate dihydrate constitutes the main phase in the amount of 91.4 % before and 96.8% after the process of flocculation. There are two major endhotermic effects on DTA curves which corresponds to the evaporation of water.

Amplitude of the first endothermic effect at 146 ^oC (Fig. 6a) and 147 ^oC (Fig. 6b) is higher than the second one, which is observed at 173 ^oC (Fig. 6a) and 172 ^oC (Fig. 6b). At the temperature range of 340-420 °C with the maximum at 370 °C, anhydrite $\beta CaSO_4$ (III) crystallized and formed anhydrite $\beta CaSO_4$ (II) with relevant exothermic effect.

For both waste gypsum samples (Fig. 6a and b), a shift at the beginning of the dihydrate dehydration process is visible. It can be noted, that the habit of crystals is responsible for the shift of the beginning of dehydration towards lower temperatures, whereas pollutants and flocculant have a significantly lower impact. There is no difference in the dehydration process, since endothermic effects of waste gypsum before and after the flocculation process are comparable. This may prove that both pollutants and flocculant are individual phases and they should not interrupt the course of the waste dehydration process. High purity of the waste containing 96.8% $CaS0_4 \cdot 2H_2O$ results from separation of general and usable drains from technological drains.

X-Ray Diffraction (XRD) analyses of waste gypsum samples before (Fig. 7a) and after the process of flocculation (Fig. 7b) indicate that the content of anhydrite in the sample b is lower than in the sample a. It is a result of its long lasting contact with water (Fig. 7).

The closed water circuit causes long lasting contact of gypsum particles with growing content of flocculant. It results in minor changes of the waste structure. The SEM images of waste gypsum before and after the process of flocculation are shown in Fig. 8. The large quantity of flocculant causes restabilization of aqueous liquid waste (sample b). It is a result of a large electrical charge, originating from ionic functional group of the polymer, occurring on the surface of particles.

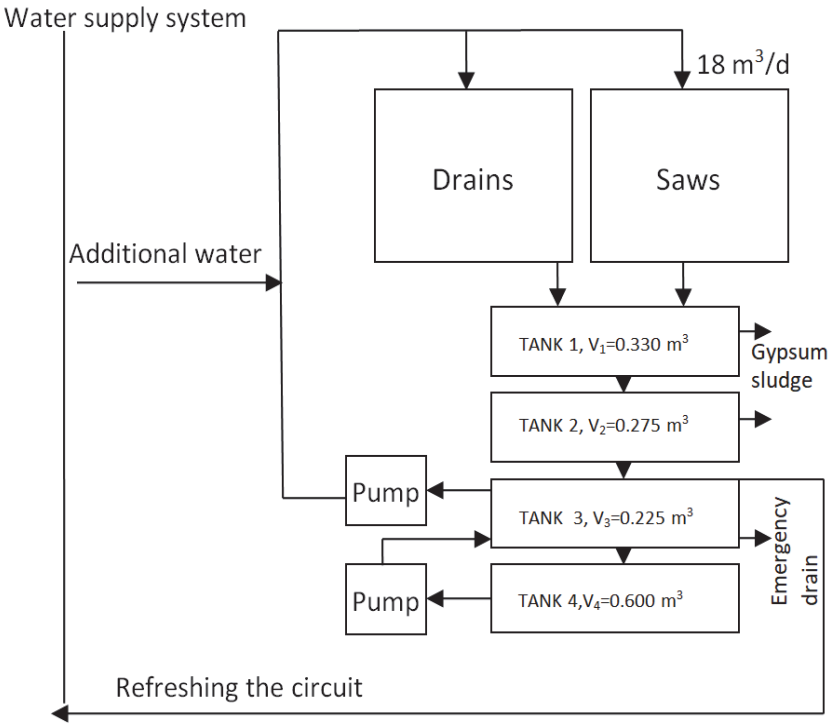


Fig. 4. The proposed block diagram of the water–sewage for machining of casts in Ortolab SA facility – with water circuit closure

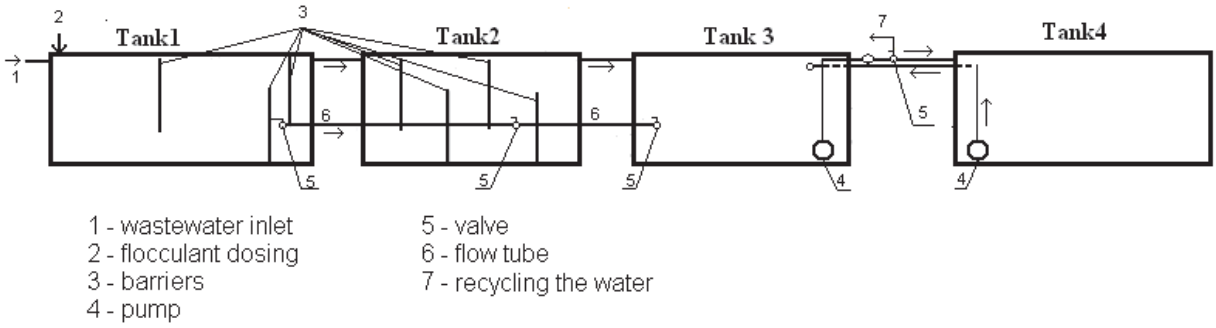


Fig. 5. The scheme of the technological system of gypsum separation from the gypsum wastewater

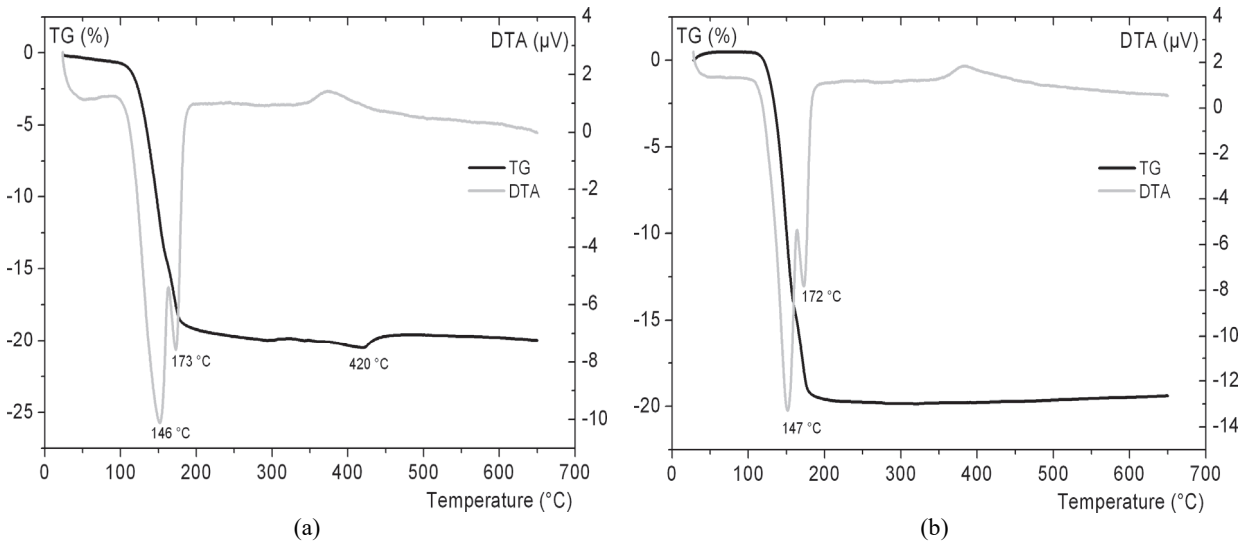


Fig. 6. TG/DTA of waste gypsum sample before (a) and after (b) implementation of technology (conditions: 25÷1000 °C, 10 °C/min, air)

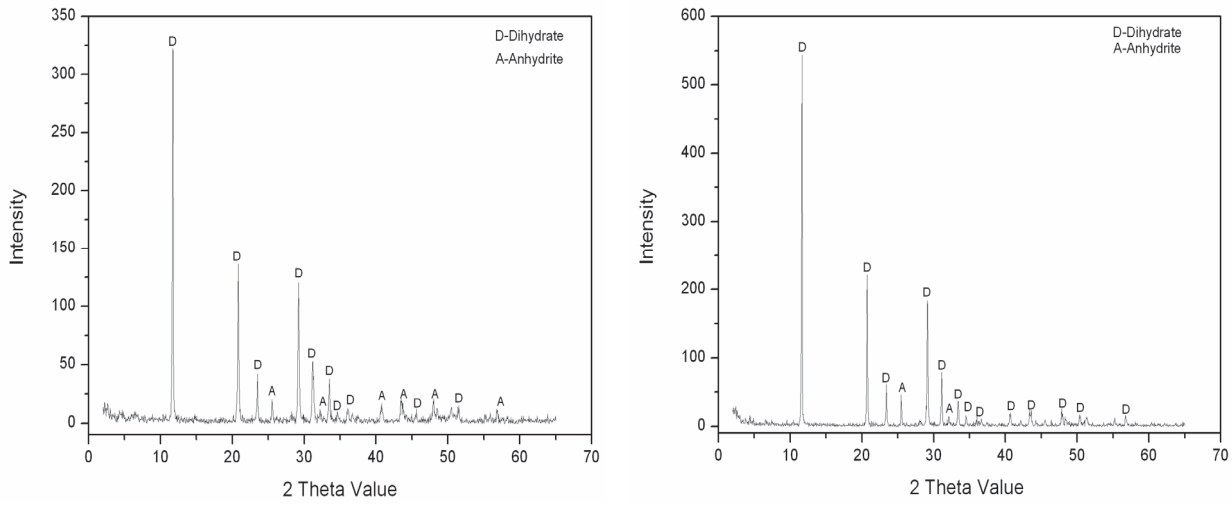


Fig. 7. XRD patterns of waste gypsum sample before (a) and after (b) implementation of technology

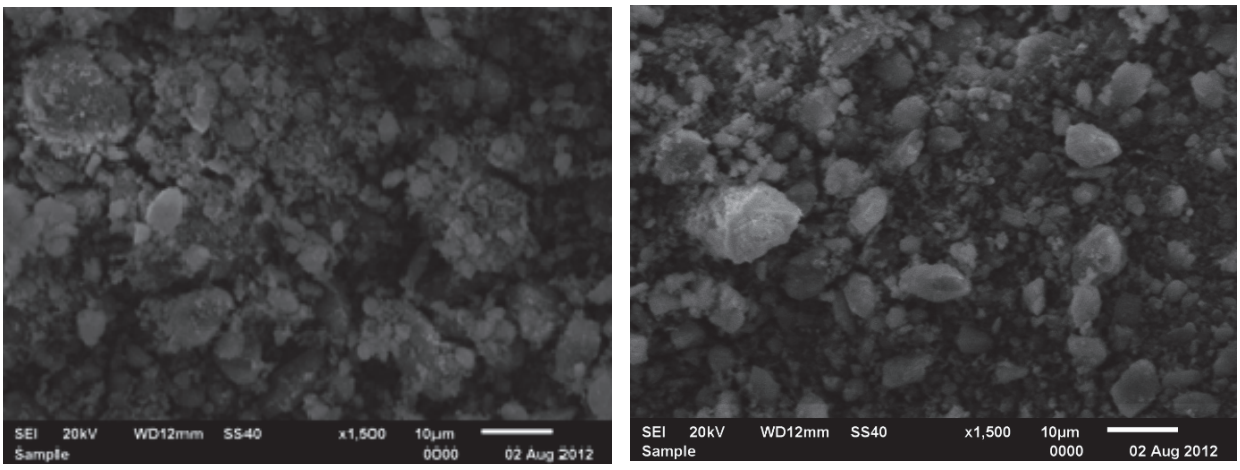


Fig. 8. SEM images of waste gypsum sample before (a) and after (b) implementation of technology

In order to recognise gypsum waste properties, resulting from the system (Fig. 5), gypsum mixtures with the addition of building plaster, cement, and activators have been blended. The process water with high levels of sulphates and flocculant was used. Obtained results were compared with dehydrated gypsum and calcined at the temperature of 160 °C. The ratio of w/g, setting time and compressive strength have been defined.

In attempts to solidify waste gypsum in the first method (without the dehydration process), standard mixtures have been prepared. The diameter d of standard cake spreading is 180 mm. The composition of mixtures and calculated w/g ratio with water content in waste gypsum, and powder density of hemihydrate and calcium sulphate dihydrate is indicated in Table 1.

Mixture B1 is exclusively composted of building plaster. The ratio of w/g is 0.6. It is in compliance with building plaster standard. Mixture S1 is composed of waste gypsum; w/g is high (1.33). High w/g ratio is caused by the flocculant, which changes liquid viscosity. Content of water with a flocculant within 43% in the waste gypsum increases viscosity and causes setting force growth of particles in the flocculated material. Implementation of increasingly larger doses of building plaster in mixtures between SB20 to SB50 has little effect on the w/g (0.90 to 1.07). Dilution of water with a flocculant in make-up water $w_z/w_{f,}$ with respect to mixtures between SB20 to SB50, was 1.63; 2.2; 2.9; 4.4, respectively. Dilution of SB60, SB70, and SB80 mixtures was 7.0; 10.9; 23.3, respectively. The amount of added water slightly influences flocculating activity. Due to this fact, viscosity decreasing and increased cake spreading are not possible, which results in decreased quantity of make-up water. An increase in the building plaster quantity introduced into the mixtures SB60-SB80 causes the increase of medium density. The cohesion force is increasing due to a higher quantity of flocculant-covered particles. Demand for water, needed to bind a hemihydrate along with its increasing quantity, is also growing. It results in the increase of w/g from 1.2 to 1.71. SB50 mixture has a beneficial and acceptable content of waste gypsum and w/g is 1.07.

The quantity of make-up water in mixtures increase along with the increasing β factor as the relation of building plaster and waste gypsum (Fig. 9).

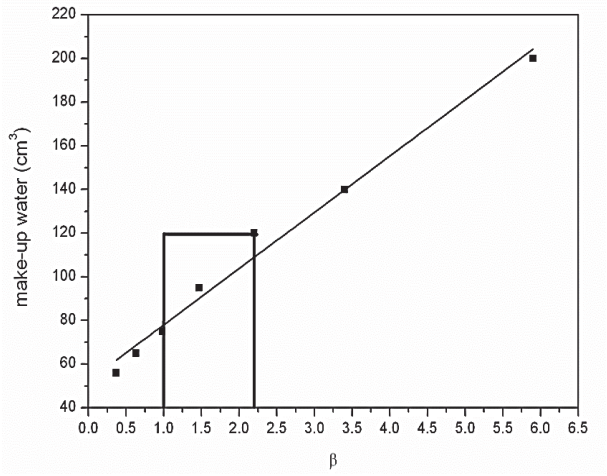


Fig. 9. The quantity of make-up water in the samples depending on the β factor

Mixtures from the circled area in Fig. 9 have been chosen in order to examine setting time. This option was chosen due to an adequately high content of waste gypsum β=1; 1.5; 2.25. Gypsum slurries have been made from the chosen mixtures and their setting time were examined (Figs. 10-11).

Examinations of mixtures' setting time were conducted with relation to their different compositions, in which variables constituted the content of activators, Portland cement, and water (Table 2). These examinations confirm the effectiveness of the activator and Portland cement introduction into the waste gypsum mixture in order to accelerate the initial and final of its setting. The usage of a smaller quantity of make-up water causes increase in concentration of activators and alkalis present in Portland cement, which significantly shortens setting time (Figs. 10-11). An optimal result of setting time examination was obtained with reference to mixture SB50" (Fig. 11). The initial of setting starts after 83 minutes and final after 300 minutes. Solidified mixture has uniaxial compressive strength after 28 days of seasoning equal to 2.85 MPa. Waste gypsum after dehydration process at 160 ^{0}C has the values of standard cake spreading d=180 mm (Table 3).

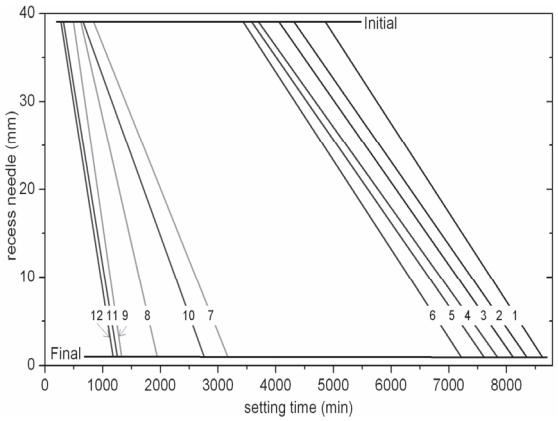


Fig. 10. Setting time of 1-12 samples

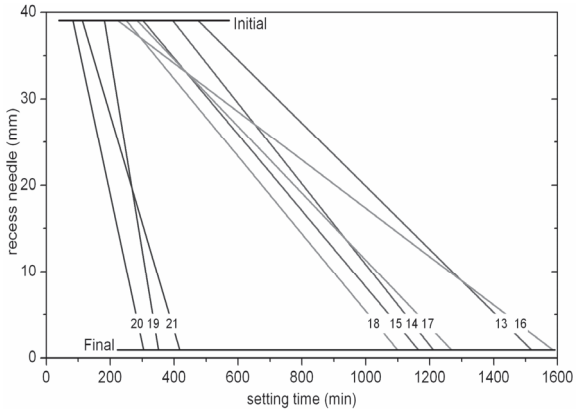


Fig. 11. Setting time of 13-21 samples

The w/g ratio of gypsum mixtures after dehydration is slightly smaller than in the case of gypsum not subjected to this process (Table 4). The setting time is significantly shorter and its value corresponds to gypsum from the manufacturer (10-14 min). The compressive strength of the solidified gypsum mixture after dehydration at 160 ^{o}C is 8.5 MPa (in the dry state). The compressive strength in the saturated water is 2.3 MPa. The value of the softening coefficient is 0.259. The results of leaching have shown no content of water-soluble salts of sodium (Na_2O), potassium (K_2O) and iron (Fe_2O_3). Content of water-soluble salts of MgO was 0.009% by weight (<0.02% by weight is recommended by *Eurogypsum*). These values are comparable to standard building gypsum.

Table 1. Fluidity, water/gypsum ratio and β- factor of the samples

Sample	***Fluidity (mm)***	***Waste gypsum (%)***	***Building plaster (%)***	***Water (%)***	***w/g***	***β****
S1	180	42.9	0	57.1	1.33	-
B1	180	0	62.2	37.8	0.6	-
SB20	180	38.6	14.2	47.2	0.90	0.37
SB30	180	31.6	19.9	48.5	0.94	0.63
SB40	180	25.8	25.2	49.0	0.96	0.98
SB50	180	19.6	28.8	51.6	1.07	1.47
SB60	180	14.1	31.1	54.8	1.20	2.20
SB70	180	9.8	33.7	56.5	1.30	3.40
SB80	180	5.4	31.5	63.1	1.71	5.90

**β= building plaster / waste gypsum*

Table 2. Composition of the samples to the setting time examination

Nr	*Sample*	*Building plaster*	*Waste gypsum*	*Activators*			*Cement*	*Water*
				$MgSO_4$	*NaCl*	*$Al_2(SO_4)_3$*		
		(%)						
1	SB40	28.89	29.45	-	-	-	-	41.66
2	SB50	31.83	21.64	-	-	-	-	46.53
3	SB60	31.67	15.54	-	-	-	-	52.78
4	SB50	31.60	21.48	0.24	0.24	0.24	-	46.19
5	SB50	31.37	21.32	0.48	0.48	0.48	-	45.85
6	SB50	30.92	21.03	0.95	0.95	0.95	-	45.20
7	SB50	30.71	20.87	-	-	-	3.54	44.88
8	SB50	29.65	20.16	-	-	-	6.84	43.34
9	SB50	28.67	19.49	-	-	-	9.93	41.91
10	SB50	30.49	20.72	0.23	0.23	0.23	3.52	44.56
11	SB50	29.25	19.88	0.45	0.45	0.45	6.75	42.76
12	SB50	27.94	18.99	0.86	0.86	0.86	9.67	40.83
13	SB50’	33.91	23.05	-	-	-	3.91	39.13
14	SB50’	32.63	22.18	-	-	-	7.53	37.65
15	SB50’	31.45	21.38	-	-	-	10.89	36.29
16	SB50’	33.65	22.87	0.26	0.26	0.26	3.88	38.82
17	SB50’	33.39	22.69	0.51	0.51	0.51	3.85	38.52
18	SB50’	32.88	22.35	1.01	1.01	1.01	3.79	37.94
19	SB50’’	38.65	26.27	0.30	0.30	0.30	4.46	29.73
20	SB50’’	38.31	26.04	0.59	0.59	0.59	4.42	29.47
21	SB50’’	37.64	25.58	1.16	1.16	1.16	4.34	28.96

Table 3. Values of standard cake spreading for the sample after dehydration at 160 ºC

Fluidity d, mm	175	180	185	195	210
w/g	0.8	0.9	1	1.2	1.5

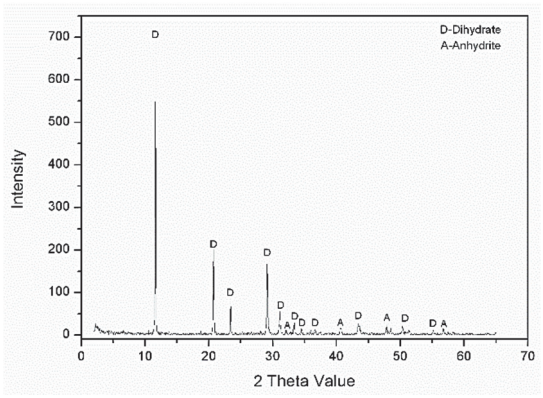
X-Ray Diffraction (XRD) analyses of solidified samples (Fig. 12) indicate a slightly higher content of anhydrite in the solidified and calcined at 160 °C sample (Fig. 12a) than in in the sample without the dehydration process (Fig. 12b).

The microscopic examination of solidified gypsum is shown in Fig. 13. As a result of these studies, it was found that the crystals size of both gypsums is similar. However, their external shape is different. The crystals of the sample b (without the dehydration) are more rounded and less porous in comparison with the sample a. Rounding, smoothing and lower porosity in the sample b may be the result of longer contact the gypsum grains in the water.

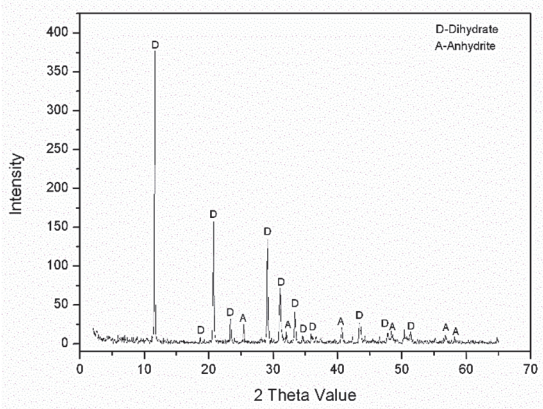
5. Conclusions

The water circuit closure solves the problem of wastewater discharge, which is characterized by over-normative content of SO_4^{-2} ions and suspensions. Periodically received hydrated waste gypsum may be managed through solidification in dehydration process or without this process. In both methods of solidification w/g ratio increase is discovered, in comparison to building plaster. It is caused by the presence of anionic flocculant.

The presence of flocculant that is bonded to the surface of calcium sulphate dihydrate particles interrupts the setting process.

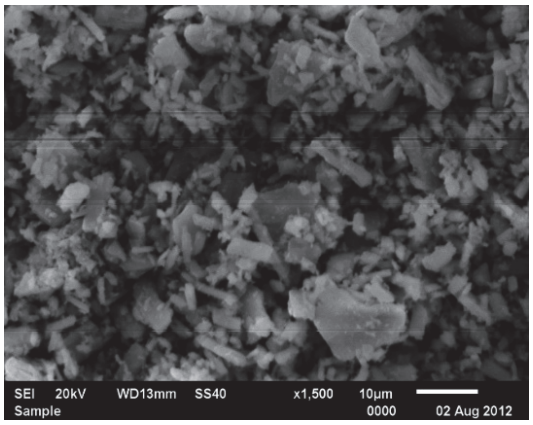


(a)

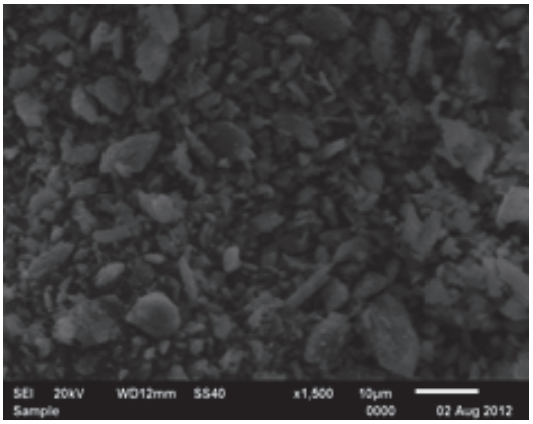


(b)

Fig. 12. XRD patterns of solidified waste gypsum sample after (a) and without (b) the dehydration in 160 ºC



(a)



(b)

Fig. 13. SEM image of solidified waste gypsum sample after (a) and without (b) the dehydration process

Table 4. Setting time for the following w/g ratio for the sample after dehydration at 160 ºC

w/g	*Recess needle, Mm*	*Time, min*
0.8	40	0.0
	39	4.0
	36	7.2
	30	9.2
	15	10.4
	1	11.5
0.9	40	0.0
	39	3.3
	36	12.0
	32	13.0
	25	14.0
	5	15.0
	1	16.0
1	40	0.0
	39	3.0
	36	12.3
	25	14.0
	20	15.0
	5	16.5
	1	17.5

Even small addition of waste gypsum into building plaster (mixture SB80) causes a lack of setting properties of the obtained mixture. After the implementation of Portland cement, building plaster and activators the mixtures acquire these properties. An optimal result of setting time examination was obtained with reference to mixture SB50".

Obtained binder has uniaxial compressive strength equal to approximately 2.8 MPa and it cannot be a substitute of building plaster. Longer setting time after the implementation of activators and lower than compressive strengths standard values make it possible to use the mixtures with activators in screed floors and roughcasts.

Solidified gypsum in the second method after the dehydration process at 160 ºC has standard value of compressive strength (8,5 MPa) and setting time for GB-D-6MPa and GB-D-8MPa fine building plasters. Solidified gypsum may be used in the production of building components.

However, it requires additional research. Nevertheless, the solidification of gypsum materials, whose particles are covered with flocculant along with water containing considerable amount of Ca^{+2} and SO_4^{-2} ions, has environmental benefits. Wastewater containing over-normative amounts of sulphates is not discharged. It is an energy-saving technology, since the dehydration process of calcium sulphate is not necessary. It solves the transportation problem of gypsum slurry and enables recycling of solidified binders or its safe storage.

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