Chapter 7 Thermal and washing treatment of South African phosphogypsum

7.1 Introduction

The impurities that should be removed or neutralised, to be able to use phosphogypsum for the control of cement hydration, are mainly water-soluble phosphorous compounds, water-soluble fluoride, and phosphorous substituted in the gypsum crystal lattice. Only the effect of phosphorous impurities on the properties of Portland cement was investigated in this study.

Ölmez and Yilmaz (1998) successfully converted the phosphorous impurities in Turkish phosphogypsum into inactive, insoluble compounds by calcination of the sample and subsequent treatment with a milk of lime solution. Only water-soluble impurities can be removed by washing with water and milk of lime. Calcium hydrogen phosphate ($CaHPO_4.2H_2O$) substitutes in the gypsum crystal lattice to form a solid solution, because of its similar crystalline parameters when compared to $CaSO_4.2H_2O$. The HPO_4^{2-} ions exist in exchanged state with SO_4^{2-} ions in these solid solutions. If the phosphogypsum is converted to the hemihydrate form, the HPO_4^{2-} ions can be removed by a calcination process, because of the subsequent change in crystal structure that occur (Ölmez and Erdem, 1989).

Naturally occurring gypsum and phosphogypsum contain various amounts of calcium sulphate dihydrate, calcium sulphate hemihydrate and anhydrite. The ratios in which these calcium sulphate forms occur can have a profound effect on the setting behaviour of cement, because their respective solubilities in water are significantly different from one another (Strydom and Potgieter, 1999). The dihydrate form is preferred in the cement industry.

The treatment of South African phosphogypsum samples by the method developed by Ölmez and Yilmaz (1988) will be investigated in this chapter. The aim of the study will be to reduce the amount of phosphorous impurities contained in South African phosphogypsum, and to ensure that the treated phosphogypsum occurs in the dihydrate form. The effect of the treated phosphogypsum as a set retarder in Portland cement will also be investigated.

7.2 Description of the experimental conditions and results of the phosphogypsum purification process applied by Ölmez and Yilmaz

In the process applied by Ölmez and Yilmaz (1988), the phosphogypsum sample was first oven-dried at 45°C. In order to investigate favourable conditions for the purification treatment, the sample was then treated with distilled water according to the procedure described in Table 7.1.

Table 7.1 Experimental procedure followed by Ölmez and Yilmaz (1988) to remove the impurities in Turkish phosphogypsum

	Solid mass (%)	Stirring time (min)	Temperature (°C)
Step 1: Optimisation of Solid ratio	12, 14, 18, 25	5	20
Step 2: Optimisation of Stirring time	14	1, 2, 3, 4, 5, 6, 9, 12, 15	20
Step 3: Optimisation of Temperature	14	5	20, 30, 40, 50, 60, 70

Two treatment methods for inactivating the impurities in phosphogypsum were used. The first method involved the neutralisation of the water-soluble impurities in the phosphogypsum with a 0.4% milk of lime solution. The untreated phosphogypsum sample was mixed with the milk of lime solution in a solid mass percentage of 14%, and was then stirred for 5 minutes at 20°C.

The other method claimed to remove the phosphorous substituted in the gypsum crystal lattice by means of thermal treatment. Untreated phosphogypsum was calcined at 130, 135, 140 and 150 °C for 5, 10, 15, 30 and 60 minutes respectively. The sample was then stirred in a 0.4% milk of lime solution for 5 minutes at 20°C.

The results obtained by Ölmez and Yilmaz (1998) are summarised in Table 7.2.

Table 7.2 The percentages of phosphorous impurities in untreated and treated Turkish phosphogypsum samples, as reported by Ölmez and Yilmaz (1998)

	Untreated	**Washing with water**	**Washing with milk of lime**	**Calcination and washing with milk of lime**	
				130 °C	150 °C
Water-soluble	0.25	0.07	Nil	Nil	Nil
In crystal lattice	0.51	0.51	0.51	0.20	Nil
Water-insoluble	0.26	0.26	0.51	0.82	1.02
Total	1.02	0.84	1.02	1.02	1.02

The results in the table indicated that all the water-soluble phosphorous impurities in phosphogypsum were converted into insoluble matter by washing it with milk of lime. There was no change in the amount of phosphorous impurities in the gypsum crystal lattice by washing the sample with milk of lime. By washing the sample with water, similar results were obtained. All the water-soluble and co-crystalline impurities were converted into insoluble compounds when the Turkish phosphogypsum samples were treated thermally followed by neutralisation with a milk of lime solution.

7.3 Experimental

7.3.1 Samples

Phosphogypsum was obtained from two South-African phosphoric acid producers, Omnia and Kynoch. Batch 1 of both of the Kynoch and Omnia phosphogypsum was used in this study. Samples were dried overnight at 45°C. Pure chemicals were obtained from Merck.

7.3.2 Instrumental analysis

Thermal analysis, Infrared analysis and X-ray fluorescence analysis were done as described in Chapter 5.2.

7.3.3 Surface area determinations

A Micromeritics Flowsorb II 2300 BET instrument was used to determine the surface areas of the gypsum samples. Nitrogen gas was adsorbed at low temperatures achieved by liquid nitrogen.

7.3.4 Cement tests

All cement control tests were performed according to standard methods described by the South African Bureau of Standards (SABS EN 196-1). The phosphogypsum samples as well as natural gypsum (used as reference) were interground with Hercules clinker. The SO_3 contents of the clinker and of each gypsum sample was first determined by a wet chemical analysis method, and each of the gypsum samples was then interground with the clinker to achieve a final SO_3 content of 2.3% in the cement. The sample was then milled to a fineness of approximately 3200 cm^2/g Blaine surface area. The relative density, initial and final setting time and compressive strength of the cement after 2, 7 and 28 days were then determined.

The SO_3 content of the gypsum samples was determined by a PPC internal standard test method, SR3 (1999): "Determination of gravimetric sulphate (SO_3)". In this method the sulphate ions are released by dissolving the gypsum in hydrochloric acid, and are then precipitated at the boiling point by a solution of barium chloride at a pH of 1.0-1.5. The SO_3 content of the solution is then determined gravimetrically.

7.3.5 Experimental procedures followed to optimise the thermal and washing treatments for South African phosphogypsum

The method used by Ölmez and Yilmaz (1988) was applied to both the Omnia and Kynoch phosphogypsum. The purpose of the treatment was to investigate its effect on the total amount of phosphorous impurities contained in South African phosphogypsum, and to study the hydrated form of calcium sulphate obtained from the treatment. The process was adjusted for South African phosphogypsum, and entailed the optimisation of the following three individual steps:

1. The phosphogypsum sample was washed with distilled water to determine the optimum solid to liquid ratio, by means of surface area analysis, at constant temperature and pressure. Solid to liquid ratios used were 10, 12, 14, 16, and 18 mass percent solid.

2. By using the optimum solid to liquid ratio, obtained from the maximum surface area, in (1), the effect of different time periods was studied stirring the phosphogypsum sample in a 0.1% milk of lime solution. (A 0.1% milk of lime solution was used due to the low solubility of $Ca(OH)_2$). The stirring times applied were 1, 5, 10, 20 and 30 minutes.

3. The effect of thermal treatment on the phosphogypsum, for various time periods, was then studied. The temperatures used were 90, 100, 110, 120, 130 and 140°C, and samples were heated at these respective temperatures for 5, 10, 20, 30, 45, 60, 90 and 120 minutes.

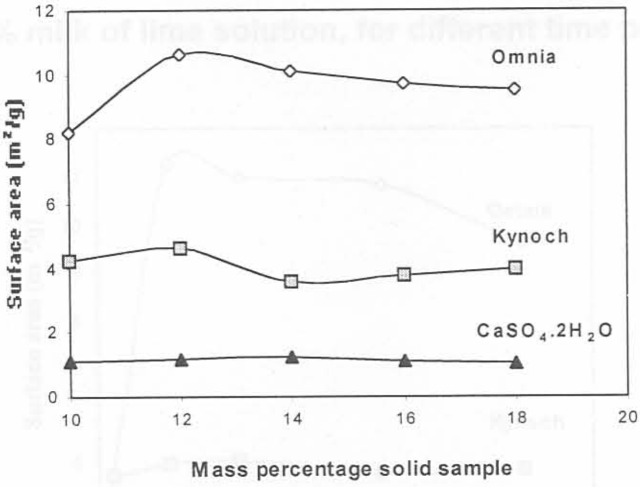
Surface area measurements were used to determine the optimum treatment condition for each step. The change in surface area can be an indication of the reactivity of the compounds, as an increase in the surface area indicates an increases in the number of surface active points and nuclei, where the reaction starts (Bamford and Tipper, 1969).

7.4 Results of the optimisation of the thermal and washing treatments for the South African phosphogypsum samples

7.4.1 Washing of phosphogypsum with distilled water

The results obtained from the surface area analyses for the washing of Kynoch phosphogypsum, Omnia phosphogypsum and $CaSO_4.2H_2O$ with distilled water in different mass percentages, are given in Figure 7.1.

Figure 7.1 Washing of the Omnia phosphogypsum, Kynoch phosphogypsum and pure $CaSO_4.2H_2O$ with distilled water in different mass percentages



Pure $CaSO_4.2H_2O$ was included as a reference. The Omnia phosphogypsum revealed a higher surface area, compared to the Kynoch phosphogypsum and $CaSO_4.2H_2O$. Both Kynoch and Omnia phosphogypsum revealed a maximum value for the surface area, and thus reactivity, when the sample was stirred in distilled water at a solid mass percentage of 12%.

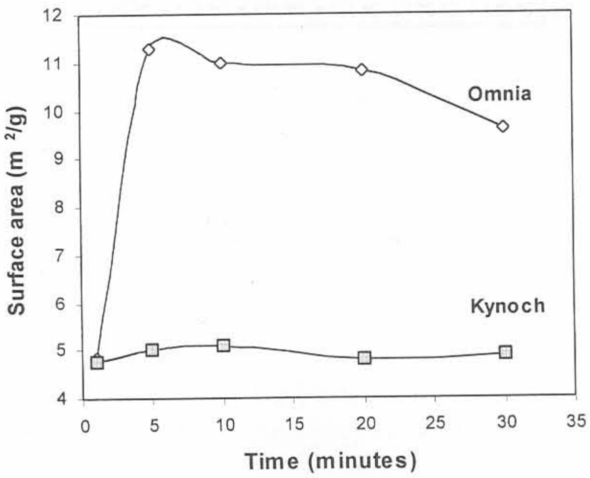
7.4.2 Treatment of phosphogypsum with milk of lime

Figure 7.2 represents the results obtained for the treatment of 12% (by mass) solid for the Kynoch phosphogypsum and the Omnia phosphogypsum samples with a 0.1% milk of lime solution. These suspensions were stirred for different time periods, after which it was filtered and dried overnight at 45°C.

The Omnia phosphogypsum revealed a significant increase in surface area, and thus reactivity, when treated with milk of lime. This increase in surface area for the Omnia phosphogypsum can possibly be explained by the hydration of calcium sulphate hemihydrate, the main constituent of untreated Batch 1 Omnia phosphogypsum. Because the untreated Kynoch phosphogypsum consists mainly of the dihydrate form of calcium sulphate, no significant increase (due to hydration) was observed in its surface area.

For the Omnia phosphogypsum, an optimum surface area was obtained after approximately 5 minutes of stirring, while it was obtained after 10 minutes for the Kynoch phosphogypsum.

Figure 7.2 Stirring of 12% (by mass) of solid for the phosphogypsum samples with a 0.1% milk of lime solution, for different time periods



7.4.3 Thermal treatment of phosphogypsum

The last step of the process involved the thermal treatment of the Kynoch phosphogypsum, Omnia phosphogypsum and pure $CaSO_4.2H_2O$ at different temperatures for different time periods. The results, given in Figures 7.3, 7.4 and 7.5, indicate that all three species reached a maximum surface area when the samples were treated between 30 - 40 minutes at a temperature of 140°C.

Figure 7.3 The thermal treatment of Omnia phosphogypsum for different time periods at different temperatures

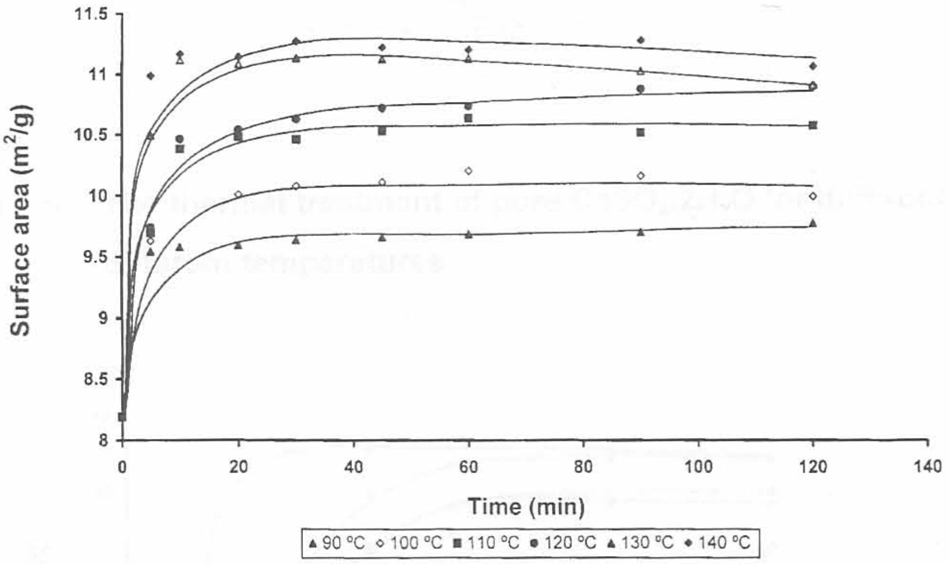


Figure 7.4 The thermal treatment of Kynoch phosphogypsum for different time periods at different temperatures

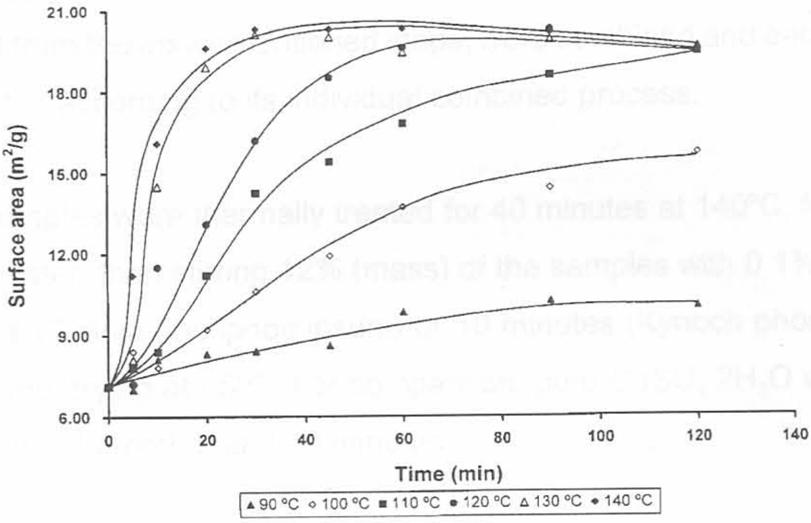
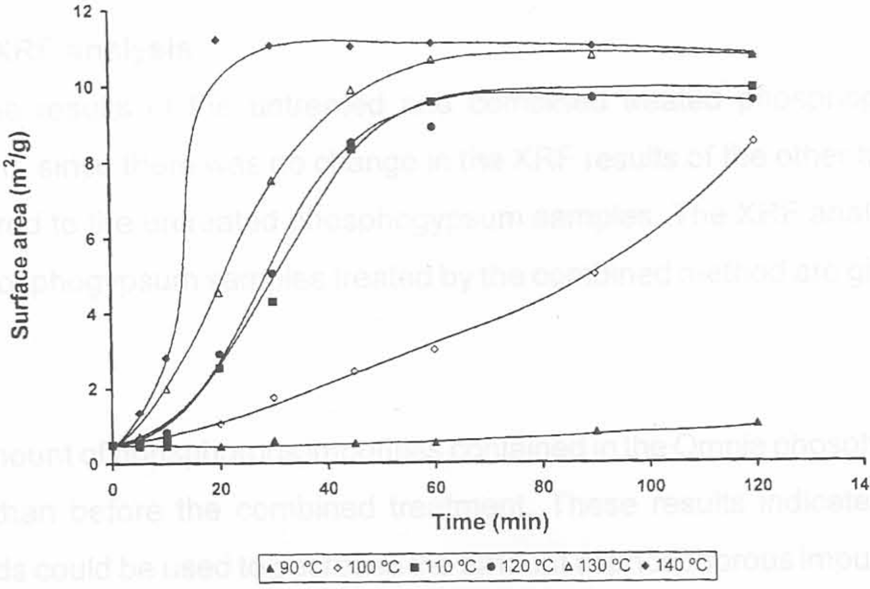


Figure 7.5 The thermal treatment of pure $CaSO_4.2H_2O$ for different time periods at different temperatures



7.4.4 Treatment of the different phosphogypsum samples by combining the washing and thermal treatment methods

The effect of combining the washing and thermal treatment of the respective phosphogypsum samples on the amount of phosphorous impurities and hydrate form of calcium sulphate was also studied. The results of the optimum treatment conditions, obtained from the abovementioned steps, were combined and each of the gypsum species was treated according to its individual combined process.

All the samples were thermally treated for 40 minutes at 140°C, followed by washing with distilled water, then stirring 12% (mass) of the samples with 0.1% milk of lime solution for 5 minutes (Omnia phosphogypsum) or 10 minutes (Kynoch phosphogypsum) and finally filtering and drying at 45°C. For comparison, pure $CaSO_4.2H_2O$ was stirred in the milk of lime solution for both 5 and 10 minutes.

7.5 Characterization of the untreated and treated phosphogypsum samples

7.5.1 XRF analysis

Only the results of the untreated and combined treated phosphogypsum samples are provided, since there was no change in the XRF results of the other treated samples when compared to the untreated phosphogypsum samples. The XRF analyses of the untreated and phosphogypsum samples treated by the combined method are given in Tables 7.3 and 7.4.

The amount of phosphorous impurities contained in the Omnia phosphogypsum was slightly lower than before the combined treatment. These results indicated that none of these methods could be used to decrease the amount of phosphorous impurities, contained in the South African phosphogypsum, significantly.

Table 7.3 The XRF results of the untreated and treated Batch 1 Omnia phosphogypsum and Kynoch phosphogypsum samples

Compounds (%)	Kynoch phosphogypsum		Omnia phosphogypsum	
	Untreated	**Combined treatment**	**Untreated**	**Combined treatment**
Al_2O_3	0.11	0*	0.04	0.02
CaO	33.04	34.57	38.38	39.80
Cr_2O_3	0	0.12	0	0
Fe_2O_3	0.09	0.25	0.05	0.06
K_2O	0	0.03	0	0
MgO	0.13	0	0.16	0.09
MnO	0	0	0	0
Na_2O	0	0	0	0
P_2O_5	**0.71**	**0.65**	**1.68**	**1.50**
SiO_2	0.16	0.18	0	0
TiO_2	0	0	0	0
V_2O_5	0	0	0	0
ZrO_2	0	0.04	0	0
Sr	0.28	0.38	0.32	0.33
SO_3	44.06	46.06	51.24	52.34
Loss on ignition	22.21	19.29	10.06	6.99
Total	100.79	101.57	101.93	101.13

*0 means that the compound was present in a quantity below the detection limit

Table 7.4 The XRF results of the untreated and treated Batch 1 Omnia phosphogypsum and Kynoch phosphogypsum samples, normalised to a loss free basis

Compounds (%)	Kynoch phosphogypsum		Omnia phosphogypsum	
	Untreated	**Combined treatment**	**Untreated**	**Combined treatment**
Al_2O_3	0.14	0*	0.04	0.02
CaO	42.05	42.02	41.79	41.43
Cr_2O_3	0	0.15	0	0
Fe_2O_3	0.11	0.30	0.05	0.06
K_2O	0	0.04	0	0
MgO	0.17	0	0.17	0.10
P_2O_5	**0.90**	**0.79**	**1.83**	**1.59**
SiO_2	0.20	0.22	0	0
ZrO_2	0	0.05	0	0
Sr	0.36	0.46	0.35	0.35
SO_3	56.07	55.98	55.79	55.60

*0 means that the compound was present in a quantity below the detection limit

7.5.2 Thermal analysis

The DSC curves given in Figures 7.6, 7.7, and 7.8, were obtained by encapsulating the samples in hermitic aluminium pans. This method resolved the two peaks, thus separating the two hydration reactions, except for the Omnia phosphogypsum which contains mainly hemihydrate.

Figure 7.6 The DSC curves for treated and untreated Omnia phosphogypsum samples in crimped Al hermetic pans

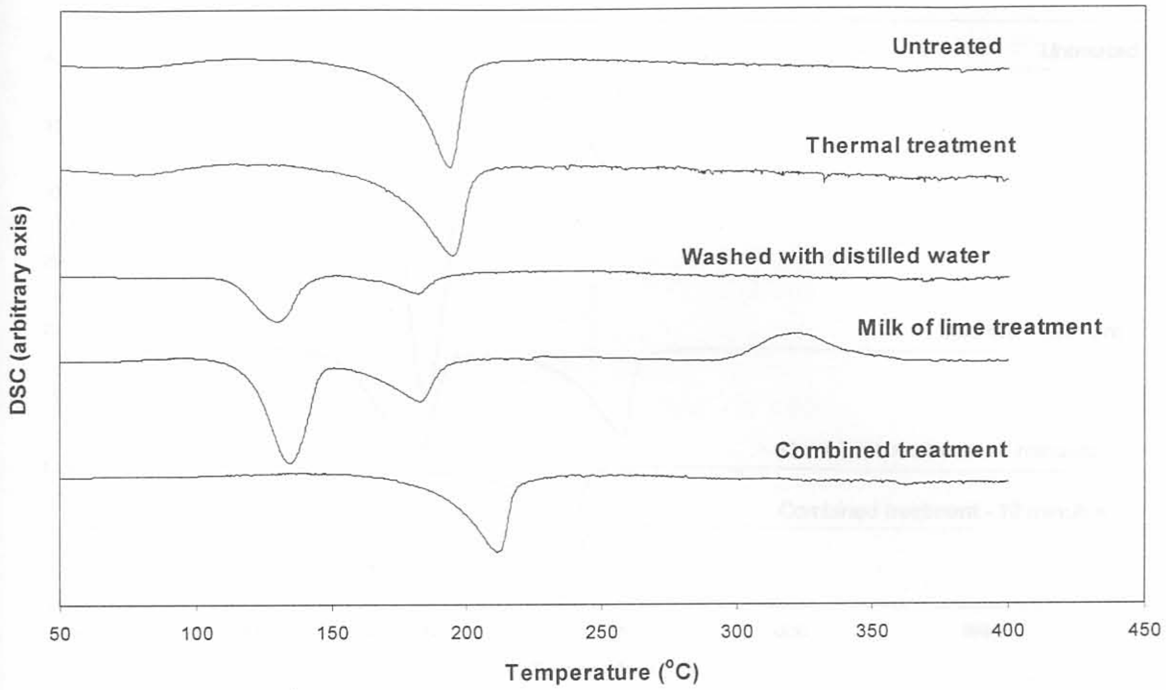


Figure 7.7 The DSC curves for treated and untreated Kynoch phosphogypsum samples in crimped Al hermetic pans

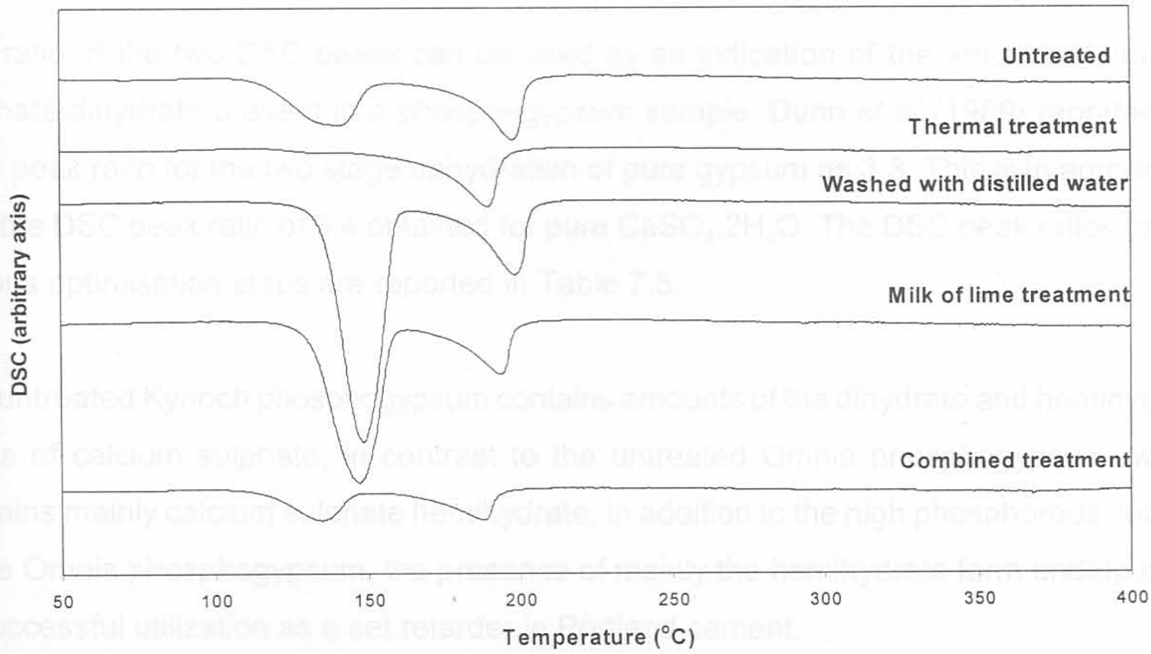
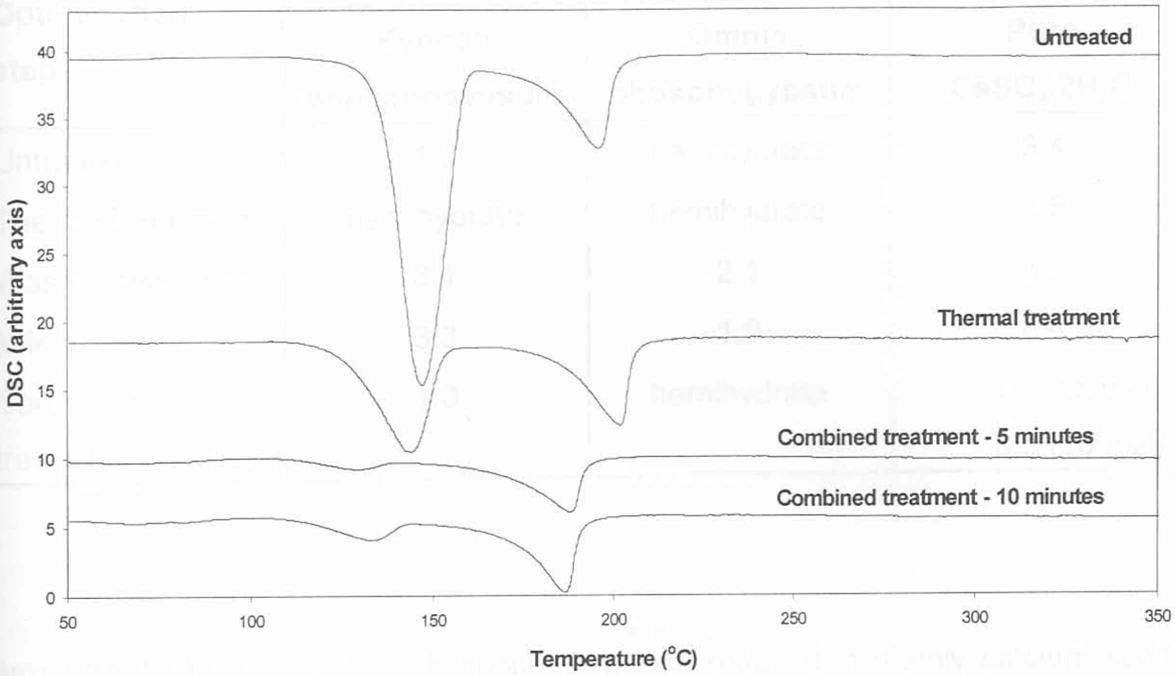


Figure 7.8 **The DSC curves for treated and untreated $CaSO_4.2H_2O$ samples in crimped Al hermetic pans**



Pure gypsum and natural gypsum are used successfully as set retarders in Portland cement. The ratio in which the different calcium sulphate species occur in the phosphogypsum, can have a major influence on its behaviour as a set retarder.

The ratio of the two DSC peaks can be used as an indication of the amount of calcium sulphate dihydrate present in a phosphogypsum sample. Dunn *et al* (1989) reported the DSC peak ratio for the two stage dehydration of pure gypsum as 3.3. This is in agreement with the DSC peak ratio of 3.4 obtained for pure $CaSO_4.2H_2O$. The DSC peak ratios for the various optimisation steps are reported in Table 7.5.

The untreated Kynoch phosphogypsum contains amounts of the dihydrate and hemihydrate forms of calcium sulphate, in contrast to the untreated Omnia phosphogypsum, which contains mainly calcium sulphate hemihydrate. In addition to the high phosphorous content in the Omnia phosphogypsum, the presence of mainly the hemihydrate form underpins its unsuccessful utilization as a set retarder in Portland cement.

Table 7.5 The DSC peak ratios obtained for the different optimisation steps

Optimisation step	DSC peak ratio		
	Kynoch phosphogypsum	Omnia phosphogypsum	Pure $CaSO_4.2H_2O$
Untreated	1.0	hemihydrate	3.4
Thermal treatment	hemihydrate	hemihydrate	1.5
Washed with H_2O	3.1	2.1	3.4
Milk of lime	3.3	1.9	3.4
Combined treatment	1.0	hemihydrate	0.1 (5 min) 0.3 (10 min)

Thermal treatment of the Kynoch phosphogypsum resulted in mainly calcium sulphate hemihydrate being formed, in contrast to pure $CaSO_4.2H_2O$, which easily rehydrates back to the dihydrate form. As expected, the Omnia phosphogypsum remained mainly in the hemihydrate form.

When washed with distilled water, the Kynoch phosphogypsum was converted to a mixture of calcium sulphate dihydrate and calcium sulphate hemihydrate as the DSC peak ratio of 3.1 indicated. The DSC peak ratio of the hydrates formed for the Omnia phosphogypsum increased to 2.1, indicating the formation of a small amount of calcium sulphate dihydrate.

Stirring with a 0.1% milk of lime solution resulted in an increase in the DSC peak ratio of the Kynoch phosphogypsum to 3.3, which is in good agreement with that obtained for pure gypsum. For the Omnia phosphogypsum a DSC peak ratio of 1.9 was obtained, which indicated that the conversion to the dihydrate form was less successful if compared to washing with distilled water.

For both the Omnia phosphogypsum and the Kynoch phosphogypsum, the DSC curves for the combined treatment are similar to that of the untreated samples. However, when pure

$CaSO_4.2H_2O$ was treated by the combined treatment, mainly the hemihydrate form is formed. A longer stirring period seemed to favour the formation of calcium sulphate dihydrate.

7.5.3 Infrared analysis

The IR spectra of the untreated phosphogypsum, treated phosphogypsum and pure $CaSO_4.2H_2O$ are given in Figures 7.9 and 7.10. The band characteristics 832 cm^{-1}, attributed to the water-insoluble phosphorous impurities, could still be observed in all the treated and untreated phosphogypsum samples.

When the IR spectra of the untreated and combined treated Omnia phosphogypsum samples were compared with that of pure $CaSO_4.2H_2O$, it was clear that these samples did not consist of only calcium sulphate dihydrate. For the treated phosphogypsum samples, a small split in the peaks at 600 cm^{-1} and 667 cm^{-1} indicated the presence of calcium sulphate hemihydrate or anhydrite. When the peaks in the region between 1600 - 1700 cm^{-1} are compared, the untreated and combined treated Omnia phosphogypsum samples showed only a single peak in comparison to the double peak of $CaSO_4.2H_2O$. This also indicated a decrease in the amount of water of crystallization present in the treated samples. For both phosphogypsum samples, the peaks between 3400 - 4000 cm^{-1}, due to the presence of water, were much narrower than that of the pure $CaSO_4.2H_2O$ samples. This confirms the results obtained by TG/DSC analyses.

The IR spectra obtained for the treated and untreated Kynoch phosphogypsum samples were similar to that of pure $CaSO_4.2H_2O$, except for the small peak at 832 cm^{-1} due to the phosphorous impurities contained in phosphogypsum. This indicated that the untreated and treated Kynoch phosphogypsum samples consisted of mainly the dihydrate form of calcium sulphate.

Figure 7.9 The IR spectra of the untreated and treated Omnia phosphogypsum samples and pure $CaSO_4.2H_2O$

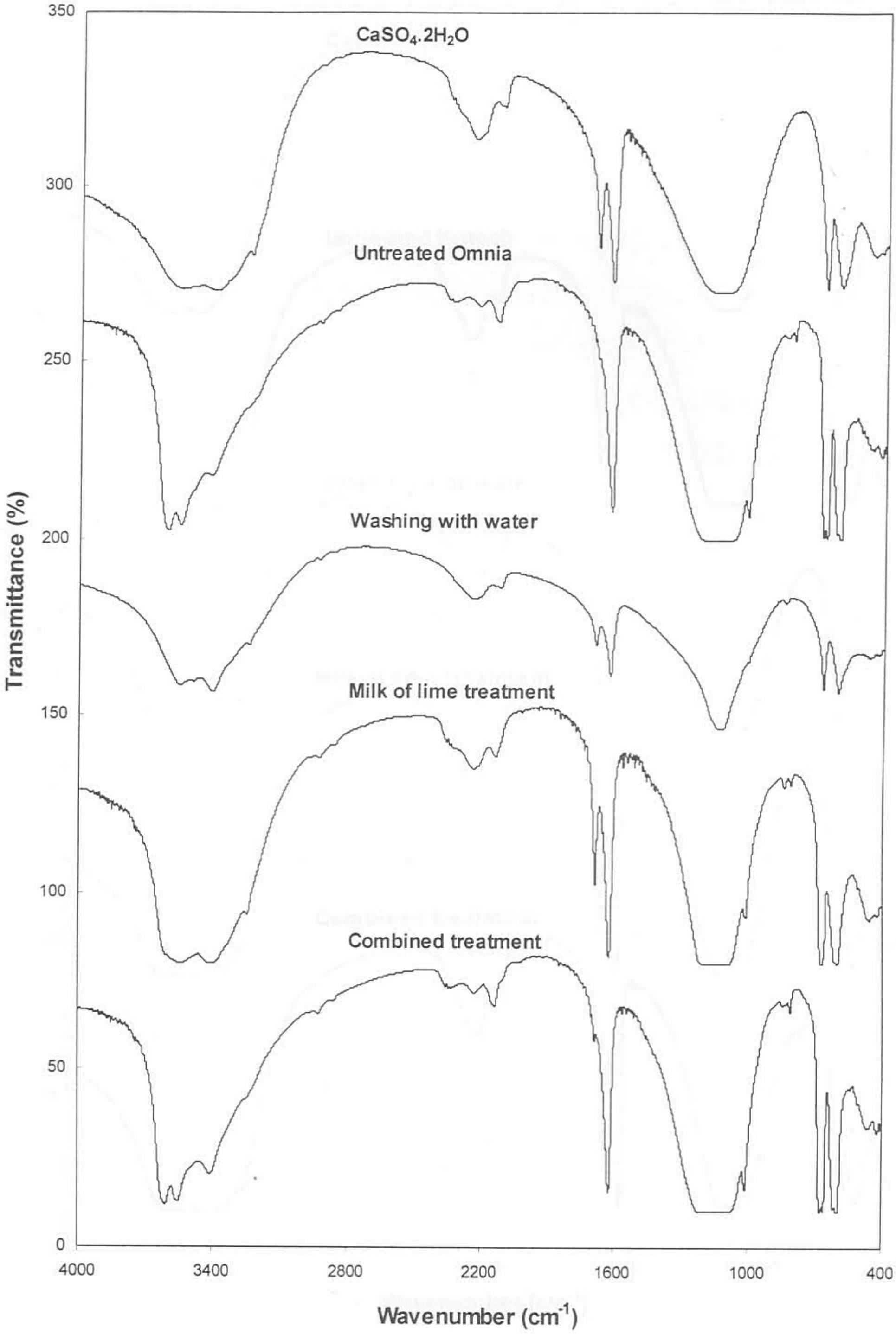
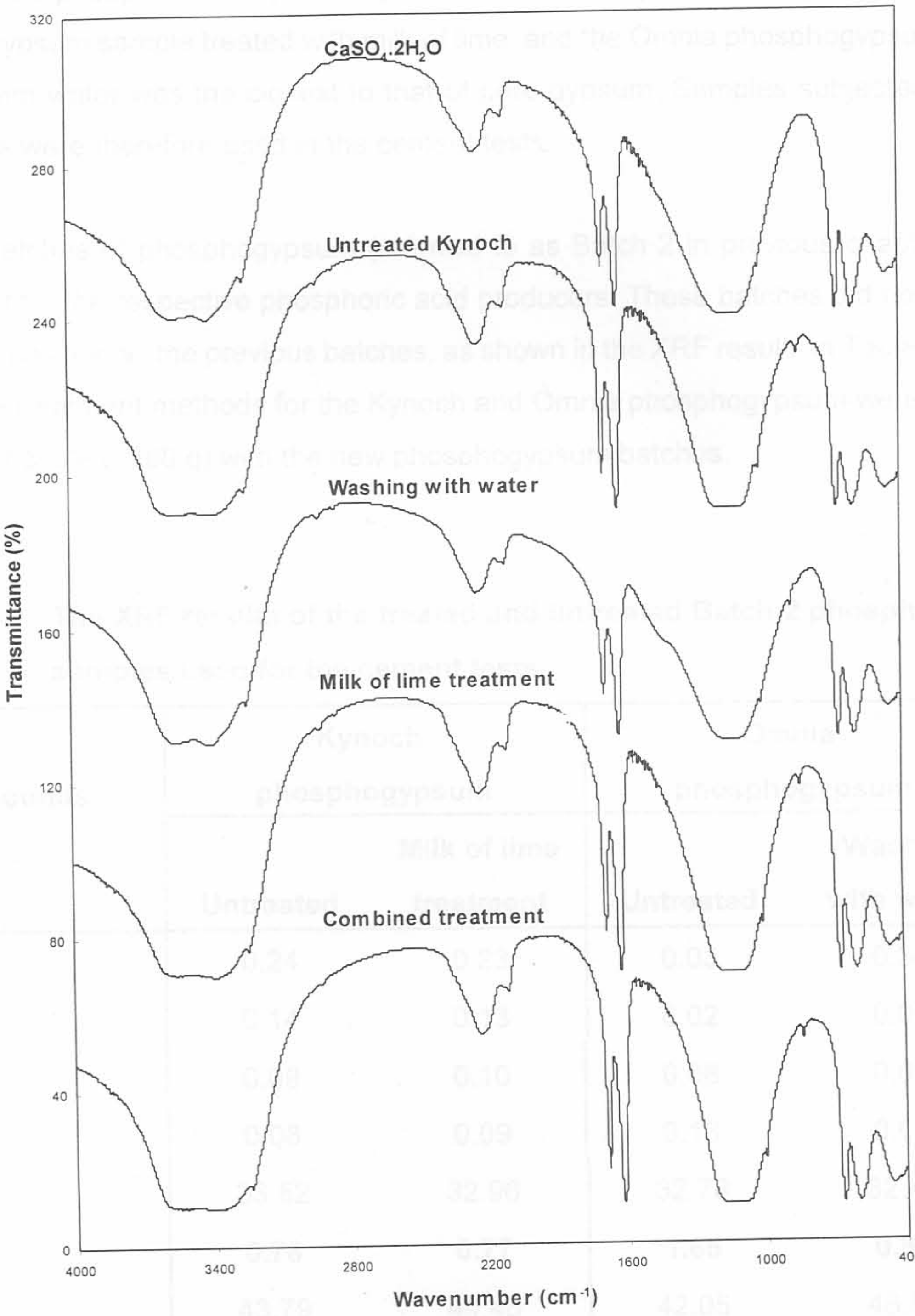


Figure 7.10 **The IR spectra of the untreated and treated Kynoch phosphogypsum samples and pure $CaSO_4.2H_2O$**



7.5.4 Cement tests

Although the thermal and washing treatment methods did not prove to be successful in removing the phosphorous impurities (Tables 7.3 and 7.4), the DSC ratio of the Kynoch phosphogypsum sample treated with milk of lime, and the Omnia phosphogypsum sample washed with water was the closest to that of pure gypsum. Samples subjected to these treatments were therefore used in the cement tests.

Second batches of phosphogypsum (referred to as Batch 2 in previous chapters) were obtained from the respective phosphoric acid producers. These batches did not have the same composition as the previous batches, as shown in the XRF results in Tables 7.6. The respective treatment methods for the Kynoch and Omnia phosphogypsum were repeated on a larger scale (±300 g) with the new phosphogypsum batches.

Table 7.6 The XRF results of the treated and untreated Batch 2 phosphogypsum samples used for the cement tests

Compounds (%)	**Kynoch phosphogypsum**		**Omnia phosphogypsum**	
	Untreated	**Milk of lime treatment**	**Untreated**	**Washed with water**
SiO_2	0.24	0.23	0.03	0.34
Al_2O_3	0.14	0.13	0.02	0.01
Fe_2O_3	0.09	0.10	0.08	0.07
MgO	0.08	0.09	0.13	0.08
CaO	33.52	32.96	32.78	32.46
P_2O_5	**0.78**	**0.77**	**1.65**	**0.87**
SO_3	43.79	44.46	42.05	46.85
Loss on ignition	20.96	21.80	22.88	21.72
Total	99.60	100.54	99.62	102.40

Table 7.7 The XRF results of the treated and untreated Batch 2 phosphogypsum samples used for the cement tests, normalised to a loss free basis

Compounds (%)	Kynoch phosphogypsum		Omnia phosphogypsum	
	Untreated	Milk of lime treatment	Untreated	Washed with water
SiO_2	0.31	0.29	0.04	0.42
Al_2O_3	0.18	0.17	0.03	0.01
Fe_2O_3	0.11	0.13	0.10	0.09
MgO	0.10	0.11	0.17	0.10
CaO	42.62	41.86	42.72	40.23
P_2O_5	**0.99**	**0.98**	**2.15**	**1.08**
SO_3	55.68	56.46	54.79	58.07

The XRF results for the untreated and treated phosphogypsum samples, used in the cement tests, are given in Tables 7.6 and 7.7. The amount of phosphorous impurities in the Batch 2 Kynoch phosphogypsum was the same as before treatment. A significant decrease in the amount of phosphorous impurities was observed when the Batch 2 Omnia phosphogypsum sample was washed with water.

The cement test results for the untreated phosphogypsum, treated phosphogypsum and natural gypsum are listed in Table 7.8. The initial and final setting times of cement containing untreated and treated phosphogypsum samples differed significantly from cement containing natural gypsum. The results indicated that both the initial and final setting times of the cement mixes was markedly retarded when the untreated Kynoch and Omnia phosphogypsum samples were used instead of natural gypsum.

This retardation effect of phosphogypsum can be ascribed to the suppression of cement hydration. The liquid phase in the cement paste generally has high alkalinity. Due to the high concentration of lime and alkali, insoluble calcium phosphate salts are produced near the cement particles soon after phosphorous enters into the alkaline liquid phase, and subsequently, they cover the surface of the cement grains, providing a protective barrier against the attack of water (Singh, 1987).

Table 7.8 **Results of the cement test performed on treated and untreated Kynoch, Omnia phosphogypsum samples and natural gypsum**

Test	**O-U**[a]	**O-T**[b]	**K-U**[c]	**K-T**[d]	**N-G**[e]
SO_3 content of gypsum (%)	47.24	46.39	46.68	45.70	42.50
Gypsum added to clinker (%)	3.46	3.52	3.50	3.58	3.85
Specific Surface of cement (cm^2/g)	3470	3175	3200	3250	3200
Relative density of cement	3.15	3.10	3.10	3.12	3.10
Initial setting time (min)	243	257	354	274	140
Final setting time (h)	5.11	5.25	6.75	5.75	3.25
2-day Compressive strength (MPa)	17.12	17.74	17.35	16.96	20.33
7-day Compressive strength (MPa)	33.91	34.15	34.41	35.36	38.04
28-day Compressive strength (MPa)	46.07	48.01	47.25	44.83	49.10

[a] Untreated Omnia phosphogypsum

[b] Omnia phosphogypsum washed with distilled water

[c] Untreated Kynoch phosphogypsum

[d] Kynoch phosphogypsum treated with $Ca(OH)_2$

[e] Natural Gypsum

There was no significant effect on the setting times when Omnia phosphogypsum was treated with water. However, the treated Kynoch phosphogypsum sample, when compared to the untreated sample, revealed a favourable decrease in both the initial and final setting times. The initial and final setting times of the cement samples containing the milk of lime treated Kynoch phosphogypsum possibly decreased due to the inactivation of some of the phosphorous impurities, and also due to the higher amount of calcium sulphate dihydrate present in the treated sample.

7.6 Conclusion

From the XRF and IR results it is evident that by applying the method used by Ölmez and Yilmaz (1988), the phosphorous impurities contained in the South African phosphogypsum samples could not be removed successfully, although some of the phosphorous impurities contained in the Batch 2 Omnia phosphogypsum could be removed partially by the washing treatment.

For the Omnia phosphogypsum, Kynoch phosphogypsum and pure $CaSO_4.2H_2O$, combination of the thermal and washing processes suppressed the formation of the dihydrate form of calcium sulphate. This combined process was therefore unsuitable for treatment of the phosphogypsum samples used.

When washing the Omnia phosphogypsum with distilled water, a DSC peak ratio of 2.1 was obtained, in comparison to the absence of calcium sulphate dihydrate before treatment. Stirring the Kynoch phosphogypsum sample with milk of lime solution resulted in a DSC peak ratio of 3.3, which is similar to that of pure gypsum.

Both the initial and final setting times of the cement mixes was markedly retarded when the untreated Kynoch and Omnia phosphogypsum samples were used instead of natural gypsum. Treatment of the Kynoch phosphogypsum sample with a milk of lime solution resulted in inactivation of some of the water-soluble phosphorous impurities, and to a higher amount of calcium sulphate dihydrate present in the treated sample. This sample revealed a favourable decrease in both the initial and final setting times when compared to the untreated Kynoch phosphogypsum samples. Treatment of Omnia phosphogypsum with water did not improve the performance of the Omnia phosphogypsum as a set retarder in Portland cement. Although the amount of phosphorous impurities contained in Omnia phosphogypsum was reduced by the washing treatment, no significant change in the setting behaviour was observed.

Application of these methods to South African phosphogypsum did not remove the phosphorous impurities successfully. Therefore, alternative methods should be investigated to reduce the harmful impurities contained in phosphogypsum.