RESEARCH REPORT

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Filtration of S-PCC slurry

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Filtration of S-PCC slurry

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Summary

Filtration properties of S-PCC and S-PCC+ different solvents were determined using Labox 100 pressure filter. Settling vessel was used to determine settling properties of S-PCC and S-PCC+ ammonium chloride. The filter cloth selected for the experiments was the tightest available (ANNE K11).

At lower S-PCC concentration (dry matter content 3.0 %) dewatering of the water slurry was significantly faster than of the slurries containing ammonium solutions. No differences appeared between the different solvents in dewatering. At higher S-PCC concentration dewatering was even slightly slower than in the case where ammonium chloride was used as solvent.

Dry matter content of cake was 47-53 % after 6 bar pumping and 5 %-units higher after membrane pressing. The dry matter (dm) content of the cake was the highest (58-60 %-units) when ammonium acetate nitrate and ammonium chloride were used as solvents. Dry matter content of cake was slightly lower when solvents were not used.

TC of the cake when ammonium acetate was used as solvent was 36 % higher than when ammonium chloride or water was used. Cl content of the cake was about 9 % when ammonium chloride was used as a solvent

The original S-PCC slurry in water with a dry matter content of 20 % settled very little during half an hour. S-PCC slurry diluted with water settled faster than S-PCC mixed with an ammonium chloride solution. 70 % of the added water was removed in settling (30 min) when S-PCC was diluted in water and 55 % when S-PCC was in a mixture with an ammonium chloride solution. Dry matter content of the liquid phase was << 1 % and solid content < 1 g/l in S-PCC settling cases. Dry matter content of liquid phase was 17 % and solid content < 1 g/l in the S-PCC+ ammonium chloride settling cases.

Confidentiality	Restricted

Jyväskylä 17.2.2014

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Preface

This filtration study was a part of the project where a new production concept to manufacture precipitated calcium carbonate (PCC) from the slag originating from Tornio steel mill is being evaluated. The study was performed in October 2013 at VTT Jyväskylä facility. Senior Scientist Pentti Pirkonen and Research Engineer Hannu Mursunen performed the research under guidance of Senior Scientist Sebastian Teir. Senior Research Technician Riitta Pöntynen did the laboratory analysis.

Jyväskylä 17.2.2014,

Pentti Pirkonen

Contents

1. Introduction

Initial process evaluation has indicated that the main part of the costs in a full-scale PCC process that uses steelmaking slag as raw material would be filtration investment costs. The filtration properties of the PCC produced from slag is unknown. Thus, filtration properties had to be determined to better evaluate the process costs. Pressure filtration was the technique chosen for water removal.

2. Goal

The goal was to evaluate dewatering properties of S-PCC and S-PCC + ammonium salt slurry.

3. Restrictions

Recently scalenohedral PCC (S-PCC) has successfully been produced in small scale laboratory experiments from steelmaking slag using the process studied here. However, the optimal process conditions for making S-PCC from steelmaking slag are still not determined. Therefore, the S-PCC used at the Veitsiluoto plant was used in the filtration experiments here. The S-PCC with a dry matter content of 20 % was supplied by Reijo Vapa.

4. Filtration experiments for S-PCC slurries

4.1 Methods and materials

Ammonium chloride, ammonium acetate and ammonium nitrate were used as solvents in 4M solution. The S-PCC dry matter (dm) content was 3.0 % and 6.0 %. The agitation time of chemicals (340 rpm) was 15 min before pressing and a temperature of 20 ^{o}C was used in the experiments.

Outotec Labox 100 PF- filter (main device, Fig. 1) and VTT filter press (Fig. 1, preliminary tests) were used in the experiments. The filter cloths studied were: ANNE T20 (air flux 1.7 L/m^{2}min), AINO K10 (air flux 0.2 L/m^{2}min) and AINO K11 (air flux 0.08 L/m^{2}min) (main cloth).

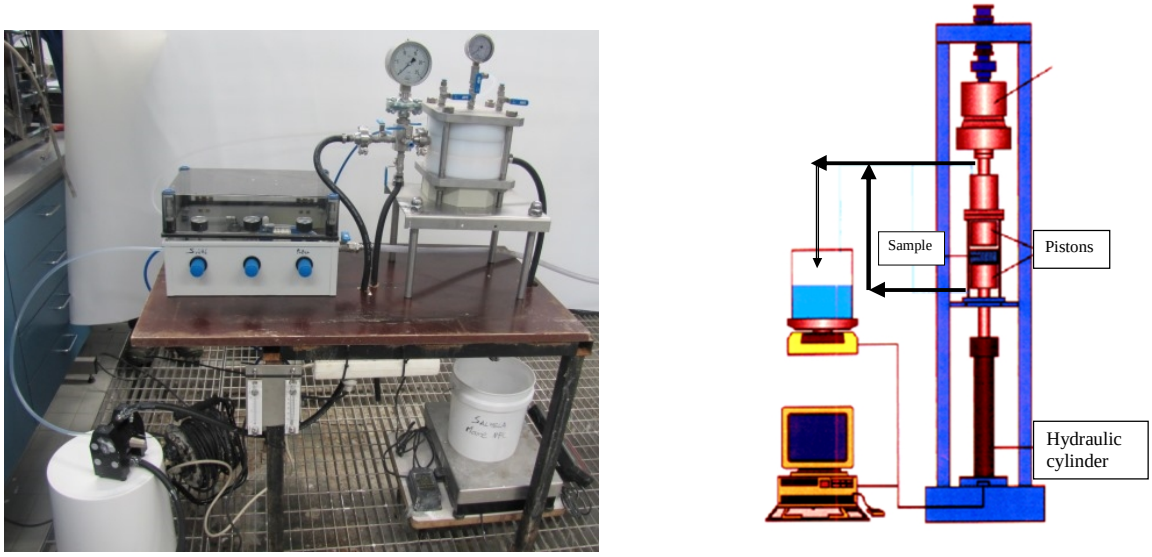
Experimental principle in pressure filtration: pressure was raised gradually in 5 minutes to 6 bars and pressing was kept in constant pressure of 6 bars as long was water came out of the cake. Then, membrane pressing in 16 bars was performed until no water came out of the cake. The cake height was 25 mm (the lowest chamber available in the press) in all the experiments.

Settling properties were determined using a standard settling vessel (Fig. 2). Settling time was 30 min or as long as settling proceeded significantly. Amount of slurry was 1000 ml in every experiment. Experimental parameters are presented in Figure 2.

Dry matter content (dm) was determined in the temperature of 105 ^{o}C. Solid content was determined using filter media of 0.2 µ pore size(SFS standard of SFS-EN872). Cake chemical content was determined as follows:

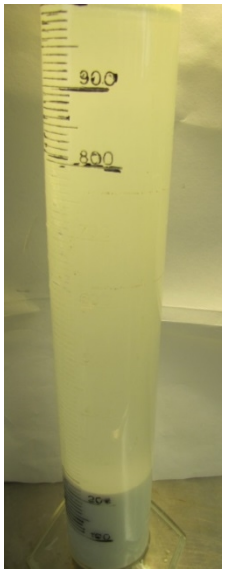
- Ca, HCl soluble

- TC, SFS-EN 13137:2001 method A
- Cl, HNO_3 soluble
- N, modified Kjeldahl method



(a) (b)

Figure 1. Pressure filtration equipment a) Labox 100 and b) VTT pressure filtration simulator.



	Vol of exp. 1000 ml	S-PCC	Extra water	NH_4Cl	Total
Exp. 0	S-PCC (dm 3,3 %)	300	1539	0	1839
Exp. 1	S-PCC (dm 20 %)	1000	0	0	1000
Exp. 2	S-PCC (dm 2,9 %) + ammoniumchlorid	300	1539	382	2221
Exp. 3	S-PCC (dm 2,9 %) + ammoniumchlorid	300	1539	382	2221
Exp. 4	S-PCC (dm 3,3 %)	300	1539	0	1839
Exp. 5	S-PCC (dm 2,9 %) + ammoniumchlorid	300	1539	382	2221

Figure 2. Settling device (1000 ml) and experimental variables.

4.2 Results

4.2.1 Cloth testing

The particle size of S-PCC used was quite small (Fig. 3) and at first a selection of filter cloth had to be tested in order to minimize the solid content in effluent. The following filter cloths were tested:

- ANNE T20 in VTT press (S-PCC + ammonium cloride); solid content in filtrate was 3880 mg/L (visible particles in filtrate)
- AINO K10 in LABOX 100 press (S-PCC + ammoniuma acetate); solid content in filtrate 570 mg/L (turbid filtrate)
- AINO K11 in LABOX 100 press (S-PCC); solid content in filtrate 7.5 mg/L (clear filtrate) (was chosen for the experiments)

Effluent contained no visible particles when AINO K11 was used as filter cloth.

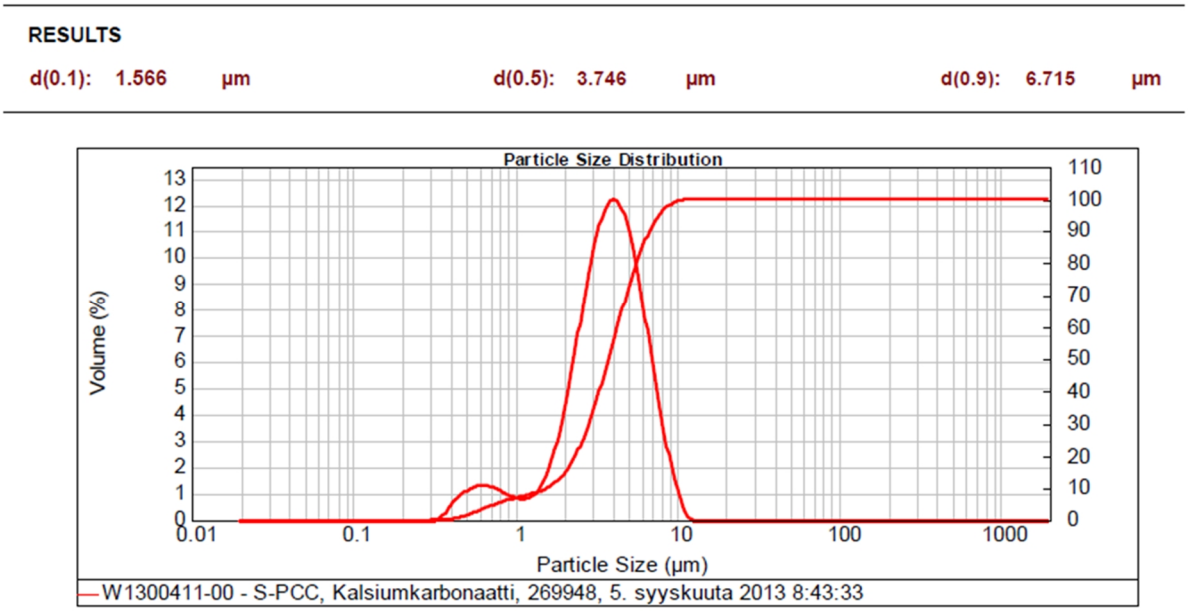


Figure 3. Particle size of S-PCC.

4.2.2 Pressure filtration

VTT filter press was at first used to see the preliminary properties of S-PCC. Due to technical problems in the filter press due to the salt solution it was decided to carry out the rest of the experiments with a Labox 100 PF-filter, which construction tolerates salt solutions better. The filter cakes produced were consistently similar (Fig. 4, left-hand picture), except in the case of ammonium acetate, where gases were apparently released from the cake during drying in the oven (Fig. 4, right-hand picture).

At the lower S-PCC concentration tested (dry matter content of 3.0 %) dewatering was significantly faster for the water slurry in comparison to the cases where different solvents were used (Fig. 5 and 6). No differences appeared between the different ammonium solvents tested. At the higher S-PCC concentration (dry matter content of 6.0 %) dewatering was even slightly slower than in the case where ammonium chloride was used as solvent (Fig. 5). Fluxes were about 2000 kg/(m^{2}h) for S-PCC and respectively about 1000 kg/(m^{2}h) for ammonium solutions (Fig 6).

Figure 4. A typical filter cake from the experiments after pressure filtration on the left-hand side and a cake produced by S-PCC+ ammonium acetate after dry matter determination on the right-hand side.

The dry matter (content of the cake was 47-53 % after 6 bar pumping and 5 %-units higher after membrane pressing (Fig. 7). The dry matter content of the cake was the highest (58-60%) when ammonium nitrate and ammonium chloride were used as solvents. The dry matter content of cake was slightly lower when only water was used. The filtrates were always visibly clear. Mass balances of the experiments were close to 97-98 %.

Some gases were released in the oven during drying of ammonium acetate. The total carbon (TC) content was higher in the cake where ammonium acetate was used as solvent, which was expected, as it is likely the carbon contained in acetate which is detected. The chloride content of the cakes produced from ammonium chloride was about 9 % (Table 1). The pH of ammonium chloride samples was 7.3, 8 in ammonium acetate sample, 7.5 in ammonium nitrate and varied between 8.3 and 8.7 in the S-PCC smaples.

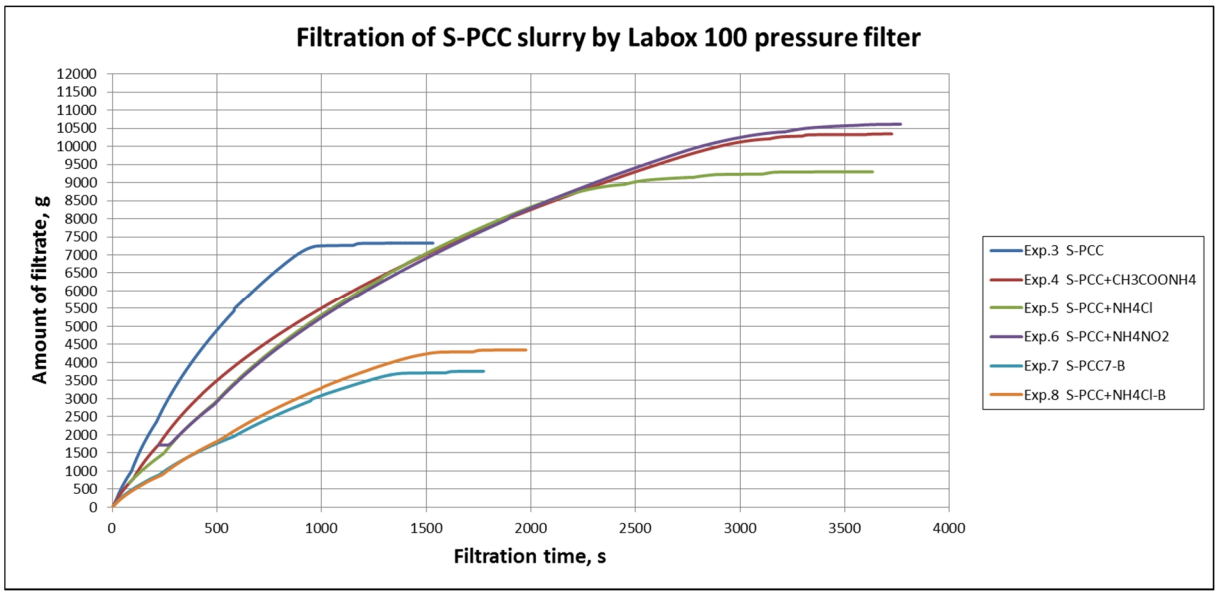


Figure 5. Filtration curves. B means S-PCC a dry matte(dm) content of 6.0 %. Other experiments were carried out in dry matter of 3.0 %.

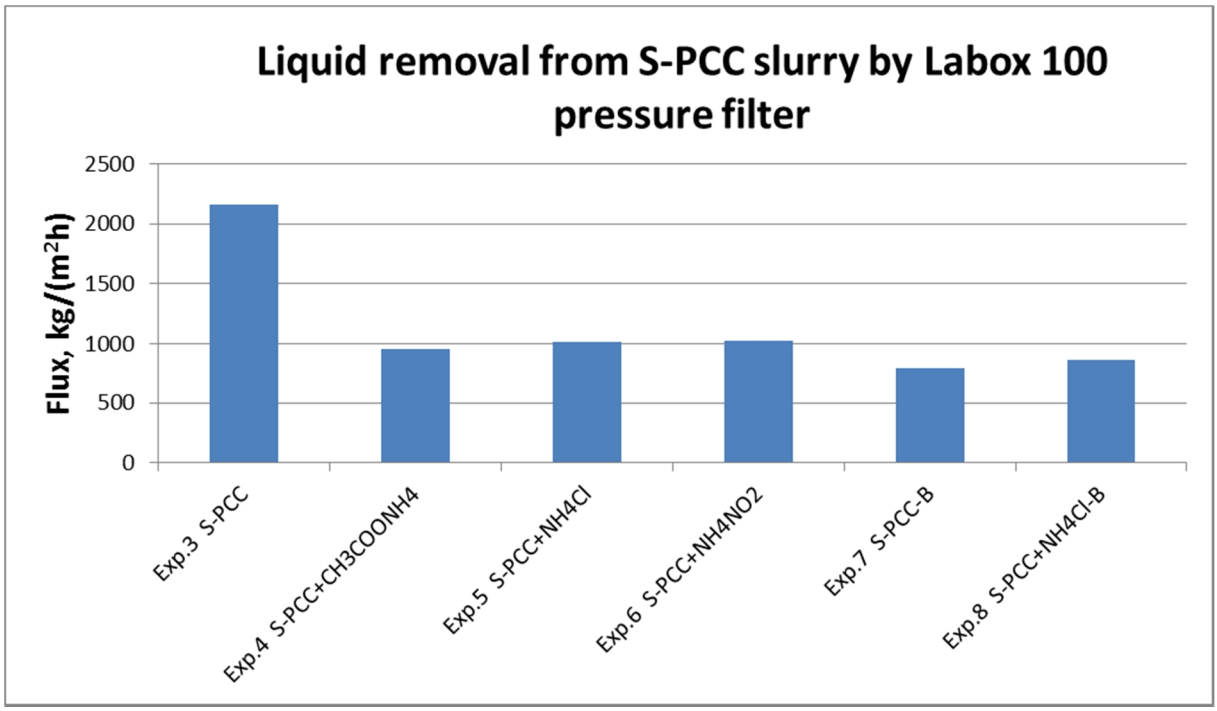


Figure 6. Water removal rate in pressure filtration. Flux was determined from the 6 bar constant pressure period in the Figure 4. B means dry matter content of 6.0 %. Other experiments were carried out in dry matter of 3.0 %.

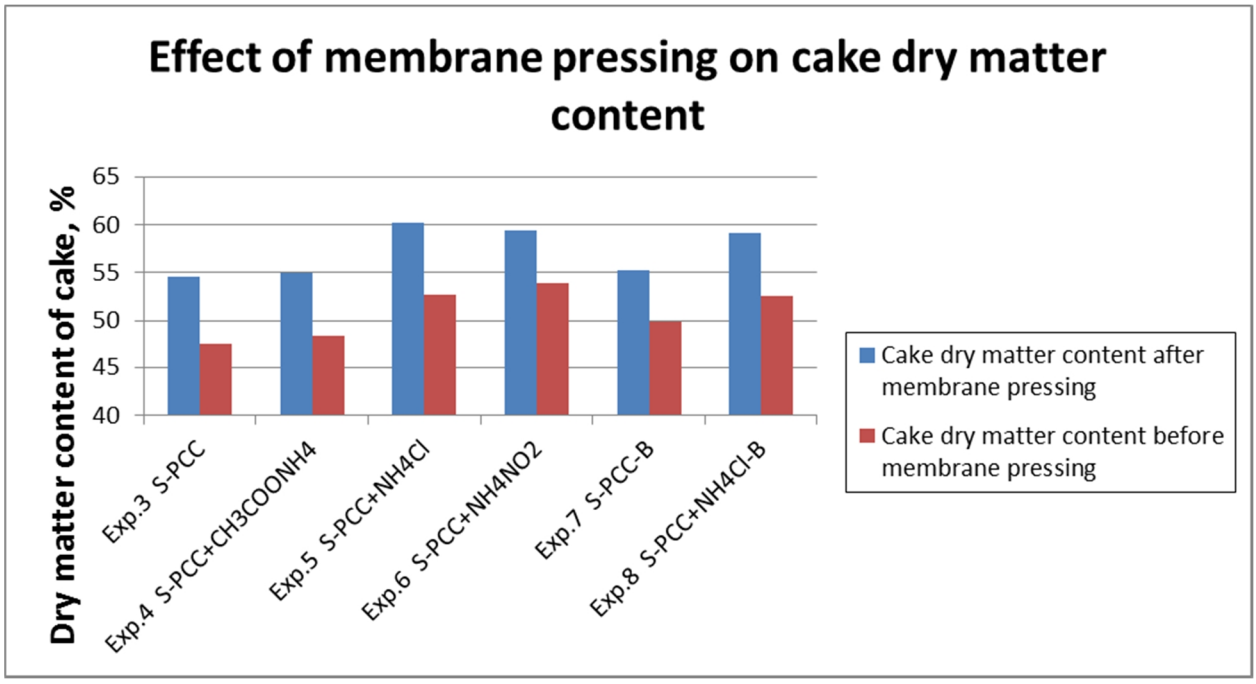


Figure 7. Dry matter content of cakes after pumping and membrane pressing phases. B means dry matter content of 6.0 %. Other experiments were carried out in dry matter of 3.0 %.

Table 1. Pressure filtration results.

Exp.	Salt	S-PCC	S-PCC	Cake	Filtrate	Filtrate	Cake	Cake	Cake	Cake
		Dry matter	Solid content	Dry matter	Dry matter	Solid content	TC	Ca	Cl	N
Nr.		%	g/l	%	g/l	mg/l	m-% dm	m-% dm	m-% dm	m-% dm
Exp. 3	S-PCC	18,3		54,6	0,3	7,5	11,2	38,9	<0.025	<0.02
Exp. 4	S-PCC+$CH_3COOHNH_4$	NA	NA	54,9	10,5	1,8	14,0	36.4	NA	NA
Exp. 5	S-PCC+NH_4Cl	NA	NA	60,3	184,3	7,1	10,1	35,1	9,1	2,7
Exp. 6	S-PCC+NH_4NO_3	18,4	206	59,4	263,8	6,2	NA	33,6	NA	<0.02
Exp. 7	S-PCC-B	18,5	203	55,3	1,5	7,5	11,2	39,3	<0.025	<0.02
Exp. 8	S-PCC+NH_4Cl-B	NA	NA	59,1	165,9	6,1	NA	35,9	8,5	NA

4.2.3 Setling properties

The original S-PCC slurry in water with a dry matter (dm) content of 20 % did not settle almost at all. A diluted S-PCC suspension in water settled a little bit better than the S-PCC suspension in ammonium chloride (Fig. 8 and 9). The settling repeated itself well (exp. 0 and 4; exp. 3 and 5).

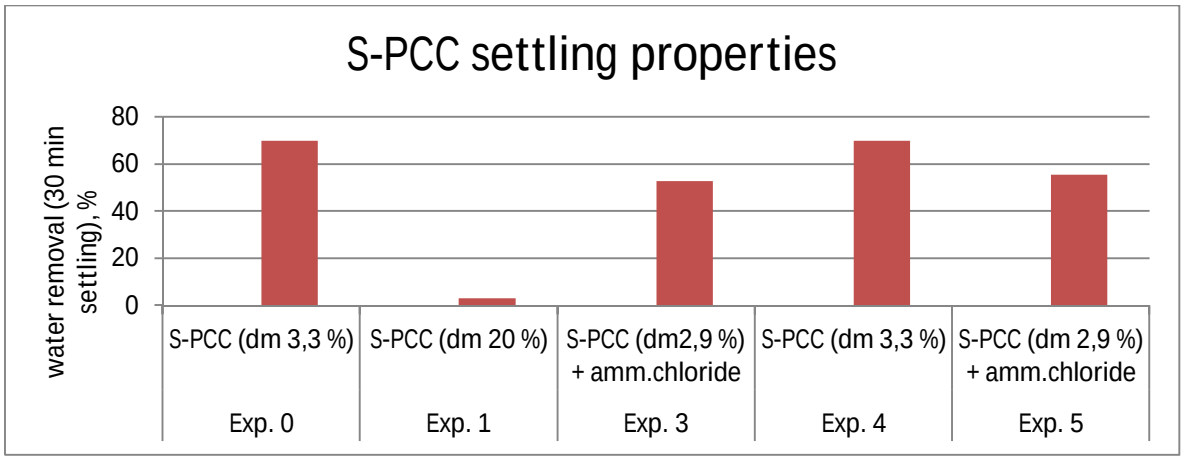


Figure 8. Amount of initial water removed in the standard 30 min settling test.

The dry matter content of the liquid phase after settling was < 1 g/l in all the cases. The ammonium chloride content in solid phase decreased slightly when settling time was incresed from 30 min to 60 min and maximum dry matter achieved was 25 %. Diluted S-PCC suspension did not release all the added water in 30 min settling (Fig. 10).

The solid content of the liquid phase was < 0.9 g/l in all the cases. Solid content in solid phase was clearly higher in S-PCC case compared to S-PCC+ ammonium chloride case. Long settling time (60 min) in ammonium experiment increased significantly solid content in solid phase up to almost same level as in the case of S-PCC (Figs. 9 and 11).

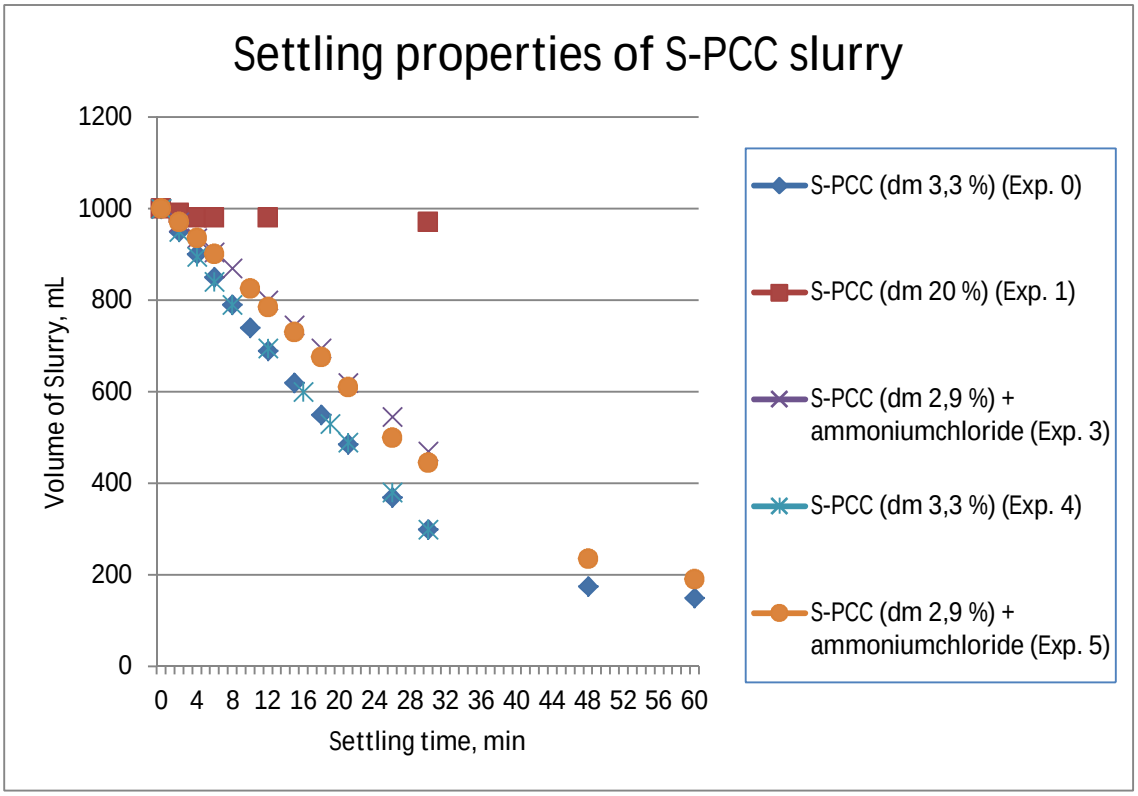


Figure 9. Settling behaviour in different cases.

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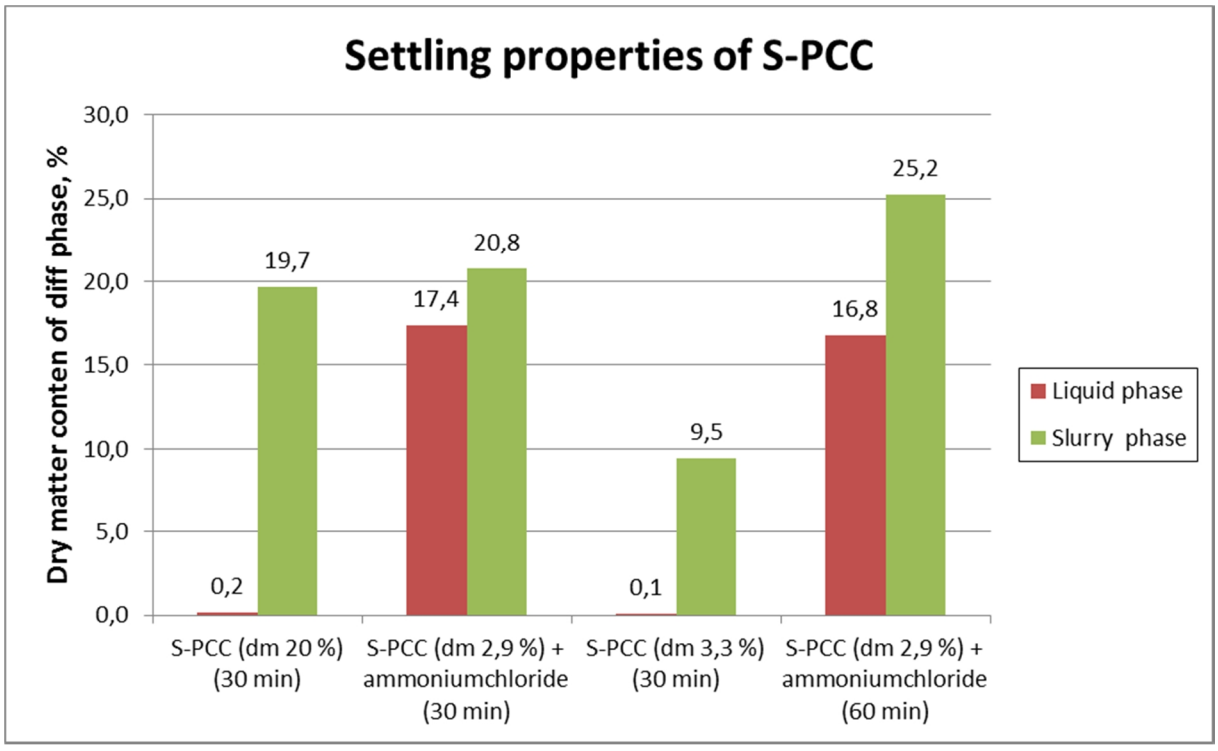

Figure 10. Dry matter content of liquid and solid phase in settling of S-PCC in different cases.

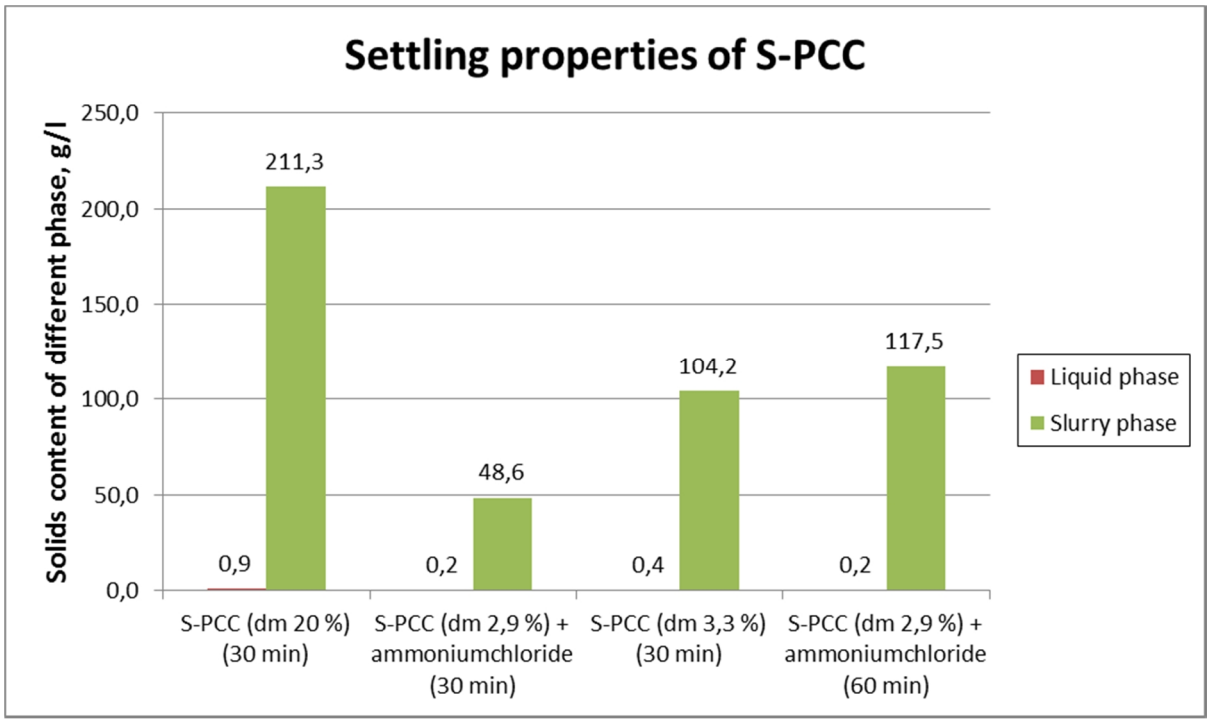

Figure 11. Solid content of liquid and solid phase in settling of S-PCC in different cases.

5. Conclusions

Following conclusion could be drawn from the study:

- No particles penetrated through the chosen filter cloth (ANNE K11).
- At lower S-PCC concentration (dry matter content 3.0 %) dewatering was significantly faster of the water slurry than of the slurries where different ammonium solvents were used.
- No differences appeared between the different solvents in dewatering properties.
- At higher S-PCC concentration (dry matter content 6.0 %) dewatering was even slightly slower of the water slurry than of the slurry where ammonium chloride was used as solvent.
- Dry matter content of cake was 47-53 % after 6 bar pumping and 5 %-units higher after membrane pressing.
- Dry matter content of cake was the highest (58-60%) when ammonium nitrate and ammonium chloride were used as solvents.
- Dry matter content of cake was slightly lower when solvents were not used. This is expected, as part of the ammonium solvents solidify upon drying.
- TC was 36 % higher in the cake when ammonium acetate was used as solvent. This is expected, as part of the ammonium acetate solidify upon drying and adds to the total carbon content of the cake
- Cl content of the cakes produced from ammonium chloride was about 9 %.

- S-PCC of original concentration (dry matter content 20%) settled very little during the experiments.
- S-PCC diluted with water settled faster than S-PCC diluted with an ammonium chloride solution.
- Dry matter content of liquid phase was << 1 % and solid content < 1 g/l in S-PCC settling cases.
- Dry matter content of liquid phase was 17 % and solid content < 1 g/l in S-PCC+ ammoniumchloride settling cases. This is expected, as ammonium chloride solidifies upon drying
- Dry matter content of solid phase was 9.5 % and solid content 104 g/l in S-PCC settling case.
- Dry matter content of solid phase was 21-25 % and solid content was 49 - 117 g/l in S-PCC+ ammoniumchloride settling cases (30 min and 60 min settling time).
- 70 % of initial water was removed in settling (30 min) when S-PCC was used and 55 % when S-PCC+ammoniumchloride was used.