Continuous separation of steelmaking slag and PCC particles from aqueous streams using an inclined settler

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Abstract

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Title:	Continuous separation of steelmaking slag and PCC particles from aqueous streams using an inclined settler
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The aim for this thesis was to investigate gravity sedimentation using an inclined settler for continuously separating particles from aqueous streams for the current two-stage steelmaking slag carbonation process producing PCC (precipitated calcium carbonate). Several experiments were conducted in different volume flows and the settler at different inclinations. For future scale-up work a dimensional analysis for efficiency of the inclined settler was performed based on the experimental results. The option of using a hydrocyclone for separation is also discussed in this thesis.

Abstrakt

Arbete:	Diplomarbete
Titel:	Kontinuerlig separering av stålslagg och PCC-partiklar från vattenströmmar med en lutande sedimentationstank
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Målet för denna avhandling var att undersöka sedimenteringsteknik med en lutande sedimentationstank för kontinuerlig separering av partiklar från vattenströmmar för den nuvarande karbonatiseringsprocessen av stålslagg för att producera PCC (utfällt kalciumkarbonat, *eng: precipitated calcium carbonate*). Experimenten utfördes i olika volymflöden och med sedimentationstanken lutande i olika vinklar. På basis av de experimentiella resultaten gjordes en dimensionsanalys över effektiviteten i separationsmetoden. Möjligheten att använda en hydrocyklon i separering diskuteras även i detta arbete.

Preface

This Master's thesis was a part of the on-going project Tekes/Cleen CCSP (2011-2015). The work was performed at the Thermal and Flow Engineering Laboratory at Åbo Akademi University during October – April 2012.

First of all, I wish to thank my supervisors Professor Ron Zevenhoven and Hannu-Petteri Mattila for their advice and guidance during this thesis work. I also wish to thank Inga Grigaliūnaitė for helping me in the beginning and Alf Hermansson for his help in the laboratory. Further I want to express my gratitude to all the other personnel at the Thermal and Flow Engineering Laboratory.

Finally I would like to thank my girlfriend, my family and friends for their support during this process.

Turku, May 2012

Sami Filppula

Swedish summary

Kontinuerlig separering av stålslagg och PCC-partiklar från vattenströmmar med en lutande sedimentationstank

Det råder ingen tvekan om att koldioxidutsläppen i världen ökar i snabb takt och drastiska åtgärder krävs. Koldioxidavskiljningstekniken (CCS) har visat sig vara ett bra alternativ för att minska på utsläppen men i dagsläget kan ingen metod anses vara både ekonomiskt genomförbar och miljömässigt hållbar. Forskning inom mineralkarbonatisering har dock gett lovande resultat. Mineralkarbonatisering innebär att kalcium reagerar med koldioxid som i sin tur bildar kalciumkarbonat i fast form. Mineralkarbonatisering har även visat sig vara ett mer säkert alternativ än de övriga CCS-metoderna.

Tekes (Utvecklingscentralen för teknologi och innovationer) påbörjade år 2005 projektet Slag2PCC (som åtföljdes av Climbus Slag2PCC Plus och Cleen CCSP) för att utreda möjligheten att karbonatisera kalciumrikt stålslagg. Projekten behandlar tre olika områden: återanvändning av stålavfall, minskning av koldioxidutsläpp och produktion av rent utfällt kalciumkarbonat (PCC) som kan säljas vidare. Projekten har varit ett samarbete mellan Åbo Akademi och Aalto-universitetet med industriella samarbetspartners som ståltillverkaren Ruukki, processtekniska företaget Outotec och gruvföretaget Nordkalk.

Fastän utvinningen av kalcium från stålslagg har gett goda resultat i tidigare laboratorie-experiment är det osäkert ifall Ruukki kommer att ändra sin ståltillverkningsprocess vilket även påverkar slaggsammansättningen. Det har rapporterats att det nya slagget innehåller mera vanadium (V) och mindre fritt kalcium än det konventionella stålslagget. Experiment har visat att utvinningsgraden av kalcium från det konventionella slagget är ~20 %, medan utvinningsgraden av kalcium från det nya slagget ligger på ~7 %. I en kontinuerlig PCC-produktionsanläggning innebär detta att från en slaggproduktion på 25 t/h kan man utvinna ungefär 5 t/h kalcium från det gamla slagget, medan man kan utvinna endast 1,75 t/h från det nya slagget.

Preliminära försök med kontinuerlig separering av partiklar med en lutande sedimentationstank ufördes innan experiment i större skala initierades. I dessa experiment användes sfäriska

Ballotini-glaspärlor och konventionell stålkonverterslagg som partiklar. Experimenten utfördes i olika volymflöden (40-65 L/h) medan sedimentationstanken var lutande i fyra olika vinklar (0°, 10°, 30° och 45°). Experimenten med glaspärlorna visade goda resultat; samtliga pärlor sedimenterades i tanken, vilket gav en separationsgrad på 100 %. Sedimenteringen visade även en markant skillnad vid lutning av tanken. När tanken var i 0° vinkel vid ~65 L/h volymflöde formades ett 70 cm långt partikelsediment längs den lutande väggen, medan det vid 10°, 30° och 45° lutningar bildades 47,5 cm, 42 cm och 40 cm långa partikelsediment. Vid det lägsta volymflödet (~40 L/h) som testades när tanken lutade i 0° och 10° vinklar blev partikelsedimenten lika långa (~20 cm). Glaspärlorna färdades längre i experimenten än antytt i teorin men det kan ha berott på det pulserande inflödet från den peristaltiska pumpen. Partikelfraktionen av glaspärlorna var lika i alla experiment (125-250 µm).

Det utfördes tre olika experiment med stålkonverterslagg. Inför experiment med sedimentationstanken lutande i 30° och 45° vinkel sållades partiklarna till en 63-125 µm fraktion, medan partiklarna vid en 10° lutning sållades till en ≤125 µm fraktion. Inflödet var ~40 L/h i alla tre experiment och underflödet reglerades till ~7 L/h. Under experimenten blockerades underflödet av slaggpartiklarna vilket gjorde strömmen väldigt oregelbunden.

Vid 30° och 45° lutningar av sedimentationstanken formades 82 cm och 80 cm långa partikelsediment. Vid en 10° lutning formades ett partikelsediment längs hela tanken (110 cm). Fastän partikelsedimentet sträckte sig till överflödets öppning noterades inga större förändringar i partikelkoncentrationen. Partikelkoncentrationen i överflödet var nästan densamma i samtliga tre experiment med stålkonverterslagg (~0,13 g/L). Partikelkoncentrationen i inflödet var ~90 g/L, vilket gav en avskiljningsgrad på 99,86 %.

I det större extraktions- och karbonatiseringsexperimentet användes det nyare stålslagg som nämndes tidigare. För separering av slaggpartiklarna och PCC-partiklarna användes även samma sedimentationstank som tidigare. I dessa experiment siktades slaggpartiklarna till en 63-425 µm fraktion, volymflödet reglerades till ~40 L/h och sedimentationstanken vinklades i en 45° lutning. Separeringen av stålslaggpartiklarna var väldigt effektiv under extraktionssteget; partiklarna formade ett 100 cm långt sediment längs tanken och partikelkoncentrationen i överflödet var

endast 0,17 g/L. Partikelkoncentrationen i inflödet var ~84,45 g/L under experimentet, vilket gav en avskiljningsgrad på 99,8 %. Analyser av överflödet visade att partiklarna var ≤ 2 μm som sedimenteras väldigt långsamt eller inte alls på grund av deras ringa storlek.

Vid karbonatiseringen separerades PCC-partiklar bättre än förväntat. Avskiljningsgraden var ungefär 97,8 % och partikelkoncentrationen i överflödet var endast 0,1 g/L. Partikelsedimentet var ungefär 80 cm långt längs tanken. Analyser med en SEM-apparat (scanning electron microscope) visade att partiklarna var ≤ 30 μm och innehöll klorid från ammoniumkloridlösningen, vilket antyder att partiklarna bör tvättas för att behålla sin renhet och sitt marknadsvärde.

För att lättare förstora sedimenteringsprocessen för framtida syften utfördes en dimensionsanalys av effektiviteten hos sedimentationstanken baserat på de experimentella resultaten. I normala fall bestäms effektiviteten av avskiljningsgraden, men på grund av "för goda" resultat (c_o = ~0 g/L) av den beaktades sedimentlängd som den avgörande faktorn. Det bör dock betonas att de resultat som presenterats i texten visar en standardavvikelse på 0,18 från de egentliga resultaten och experiment där tanken var i 0° vinkel inkluderades inte i analysen.

Ett annat alternativ för att separera partiklar från större flöden är en hydrocyklon. Hydrocykloner är väldigt lika sedimentationstankar, de innefattar inga rörliga delar och kräver väldigt lite underhåll. De är även mindre än sedimentationstankar, vilket kan anses vara en fördel vid större volymflöden. Vid ett volymflöde på 100 000 L/h skulle den avskiljbara partikelstorleken för en hydrocyklon med längden 1,8 m och diametern 0,3 m vara ungefär 20 μm, medan den avskiljbara partikelstorleken för en sedimentationstank av samma dimensioner (L = 1,8 m, d = 0,3 m) skulle vara runt 216 μm.

Experimenten som utförts för denna avhandling visade således att det är möjligt att kontinuerligt avskilja stålslagg och PCC-partiklar från en vattenlösning med sedimentation. Partiklarna färdades en betydligt kortare sträcka när sedimentationstanken lutade, vilket även förbättrade avskiljningsgraden markant. Sedimentationstanken kan dock förbättras. Den bör förses med ett filter för en bättre utfällning av kalciumkarbonater och för att garantera partiklarnas renhet.

Underflödet bör förses med en pump för att undvika blockage och för en lättare reglering av strömmen.

PCC-partiklarna innehöll små mängder av klorid från ammoniumsaltlösningen, vilket betyder att partiklarna bör tvättas före försäljning. Användningen av tvättvattnet för en kontinuerlig process bör i framtiden undersökas.

Även om det finns vissa områden inom processen som bör förbättras, har forskningen hittills gett goda resultat. Inom en snar framtid kommer kapaciteten hos denna karbonatiseringsprocess i två steg att förstoras till 100 kg/L vid Aalto-universitetet i Helsingfors, vilket antyder att projektet är på god väg att bli en fullskalig industriell process.

List of symbols and abbreviations

%-wt	Mass-based % fraction
Al_2O_3	Aluminum oxide
AOD	Argon Oxygen Decarburization
BOF	Basic Oxygen Furnace
C	Carbon
Ca	Calcium
CaO	Calcium oxide
$CaCO_3$	Calcium carbonate, limestone
$CaCO_3 \cdot MgCO_3$	Dolomitic stone
CCS	Carbon dioxide Capture and Storage
CCSP	Carbon dioxide Capture and Storage Program
CO_2	Carbon dioxide
Cr	Chromium
ΔH	Enthalpy change
EAF	Electric Arc Furnace
Fe	Iron
GCC	Ground Calcium Carbonate
H_2O	Dihydrogen monoxide, water
IEA	International Energy Agency
IPCC	Intergovernmental Panel on Climate Change
ISE	Ion Selective Electrode
MgO	Magnesium oxide
$MgCO_3$	Magnesium carbonate
Mn	Manganese
NH_4Cl	Ammonium chloride
NH_4NO_3	Ammonium nitrate
P	Phosphorus
PCC	Precipitated Calcium Carbonate
PSD	Particle Size Distribution

Re	Reynolds number
S	Sulfur
SEM	Scanning Electrode Microscope
SiO$_2$	Silicon dioxide
t	time
TC	Total Carbon
Ti	Titanium
V	Vanadium, volume
XRF	X-ray Fluorescence Spectroscopy

Table of Contents

1   Introduction

Increasing emissions from fossil fuel combustion from various industries have resulted in growing concerns about climate change and greenhouse gases. Several alternatives have been suggested to replace fossil fuels but according to the International Energy Agency (IEA) carbon dioxide emissions are expected to grow further unless a more powerful option is presented. IEA reports that although CO_2 emissions from fuel combustion had decreased by 0.5 Gt to 29 Gt CO_2 between 2008 and 2009, they are expected to reach 35.4 Gt CO_2 by year 2035 [1].

Carbon dioxide capture and storage (CCS) techniques have long been considered as a method for reducing CO_2 emissions. CCS implies separating carbon dioxide emissions from a large point source, transporting the gas to a suitable location and storing it for a long period of time (>1000 years). However, the techniques are still under development and currently none of them are considered to be both economically and environmentally feasible [3].

Mineral carbonation is a CCS-method that offers an alternative way of CO_2 sequestration. The method involves CO_2 to react with earth alkaline oxides, such as magnesium oxide (MgO) and calcium oxide (CaO), producing carbonates. These earth oxides are also found in industrial residues, such as in steelmaking slag, which gives the possibility to use waste materials for carbonation. The biggest concerns for mineral carbonation have been finding an economically viable process route for the reaction and storage capacity or use for the end product.

One task of the ongoing Cleen CCSP-project co-founded by Tekes (2011-2015) focuses on these issues. The aim for the task is utilizing steel converter slag for mineral carbonation to produce pure precipitated calcium carbonate (PCC) suitable for a commercial market, such as for paper industry. So far wet carbonation has given the best test results and ammonium salt solutions were the most promising for extracting calcium selectively from steel converter slag [2]. The process has successfully been developed towards the currently ongoing scale-up but some problems are still at hand; such as loss of solvent components and dissolution of excess carbon dioxide and (bi-)carbonates ions in the aqueous streams.

The main focus of this thesis is, however, by finding an easy and efficient way of separating the slag and PCC particles from the aqueous solution using gravity sedimentation. While a filter requires much maintenance, gravity sedimentation needs very little maintenance and gives a low shear stress on the particles, and it was therefore considered worth testing. Finding a suitable separation technique for the so-called two-step mineral carbonation project brings it one important step closer towards a continuous industry scale process. The final goal within Cleen CCSP is to process 25 t/h steel converter slag, giving ~10 t/h PCC.

First, in chapter 2, a theoretical background for the different aspects of this project is given. In chapter 3, a description of and results from a laboratory scale extraction and carbonation experiment are presented. Chapter 4 involves several separation experiments with an inclined settler using steel slag particles and Ballotini glass beads. In chapter 5, a larger scale two-step mineral carbonation experiment is described, while in chapter 6 a dimensional analysis based on all separation experiments is presented. In addition, chapter 7 presents a separation alternative using a hydrocyclone. And finally, conclusions and recommendations for future work are presented.

2   THEORETICAL BACKGROUND

2.1 Two-stage steelmaking slag carbonation

The Climbus Slag2PCC project started in 2005 at Helsinki University of Technology (later followed by Slag2PCC Plus and Cleen CCSP where ÅAU was a partner) to study and develop processes that could be used for producing calcium-based compounds for subsequent use from steelmaking slag. Steelmaking slag has been successfully carbonated in the past [4], but the direct (single-step) carbonation process approaches produce aqueous mixtures of carbonates and other by-products from which individual components are difficult to separate. An indirect or multi-step process seemed therefore a better alternative for producing marketable carbonates like PCC.

In 2007 and 2008 Eloneva et al. reported that it is problematic but possible to produce calcium carbonates from blast furnace slag and steel converter slag using acetic acid as a solvent [4]. Further research showed that ammonium salt solutions were the most promising solvents for dissolving calcium selectively from steelmaking slags prior to carbonation [5]. As for steelmaking slag, it was found that the fraction of extracted calcium was largest for steel converter slag. Also, it was found that the smaller the steel converter slag's grain size fraction, the higher the calcium extraction (partly because of a higher calcium content). In order to obtain a good calcium extraction, the grain size of the steel converter slag should not exceed 500 μm [4].
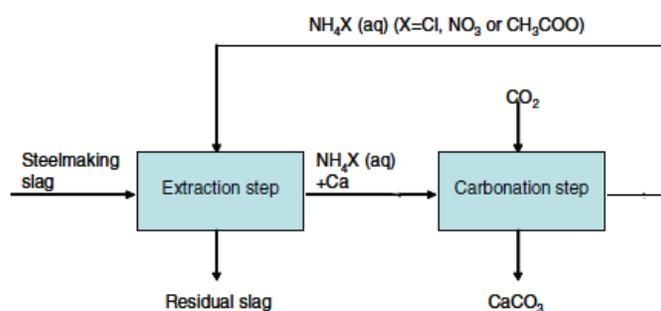
FIGURE 2.1 Process schema for production of calcium carbonate from steelmaking slag [6].

The process could be performed at room temperature and ambient pressure, and the effect of carbon dioxide pressure was limited to changes in process kinetics, the precipitation rate being slower at lower partial pressures of CO_2. In addition, if solvents with molarities higher than 1.0

mol/L or solid-to-liquid ratios higher than 100 g/L were used, iron and manganese would also extract from the steel slag, decreasing the purity and whiteness of the produced carbonates [6].

2.2 Conventional production of precipitated calcium carbonate

2.2.1 Background

Calcium carbonate ($CaCO_3$) comprises more than 4% of the earth's crust and is often found as a white mineral which occurs as chalks, limestones and marble [7]. Both natural (ground calcium carbonate, GCC) and synthetic (precipitated calcium carbonate, PCC) forms are widely used. Particularly PCC is one of the most important and versatile raw materials for use as filler in various industries, like the rubber, plastic and paper industry, due to its whiteness and chemical purity. In 2006, approximately 480 kt PCC was produced and used in Finland [8].

Calcium carbonate exists in a variety of polymorphs, of which three are crystal polymorphs; calcite, aragonite and vaterite. Calcite has a rhombohedral, prismatic or scalenohedral crystal type, while aragonite is needle-like and vaterite is spherical. Calcite is the most stable phase at room temperature under normal atmospheric pressure. Aragonite and vaterite are metastable polymorphs that readily transform into the stable phase [9]. Both calcite and aragonite are common natural polymorphs, while vaterite is rare in nature.
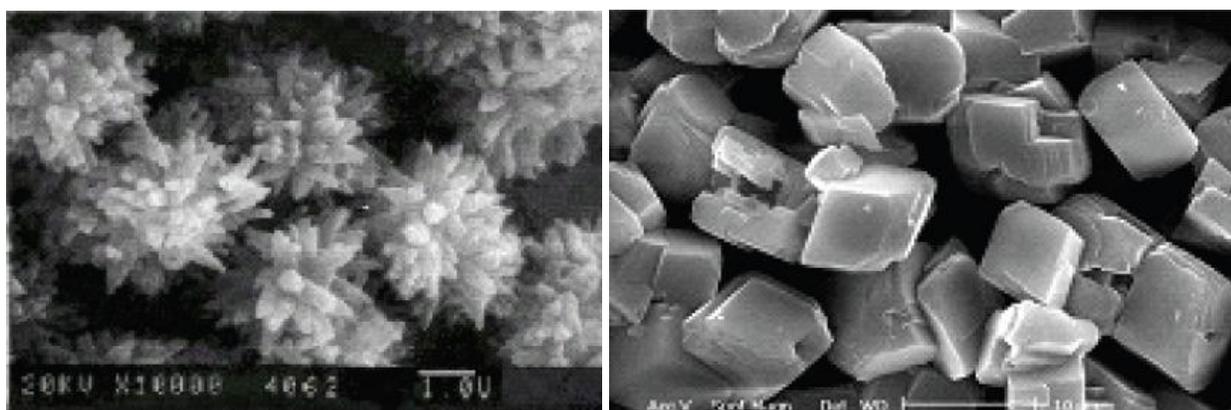
FIGURE 2.2 Different crystal types of PCC. Scalenohedral calcite (left) and rhombohedral calcite (right) [10].
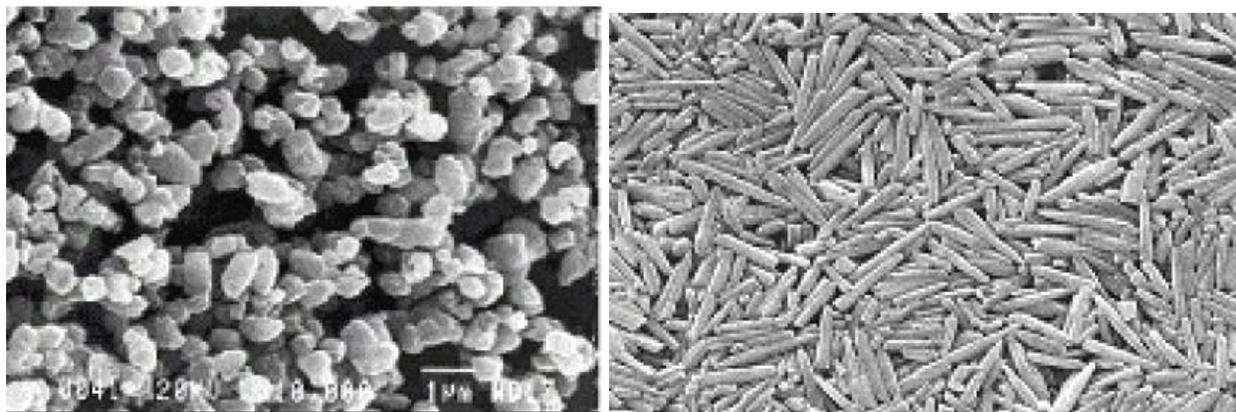
FIGURE 2.3 Prismatic calcite (left) and acicular (needle-like) aragonite (right) [10].

The purity, crystal structure and particle size of the produced PCC determine its market value. The suitability for different industrial applications is also determined by a number of parameters, like particle size distribution, morphology and specific surface area. Katsuyama et al. estimated in 2005 that the commercial price of high-purity $CaCO_3$ is about 154-269 €/m^3, while the cost of producing conventional $CaCO_3$ is about 105 €/m^3. It was also mentioned that the price of ultra-high purity $CaCO_3$ could be as high as 7 700 €/m^3 while the production cost would only be about 250 €/m^3 [11].

2.2.2 Manufacturing process

PCC is an approximately 99% pure calcium carbonate and is conventionally manufactured by a carbonation process. The PCC process is one of the most economic processes existing today.

At first, in the calcination process, the limestone is converted into calcium oxide and carbon dioxide in a kiln at temperatures above 900 ^{o}C (R2.1) [12]. Limestones are rocks containing varying proportion of calcite and dolomite with small amounts of iron-bearing carbonates. Theoretically the optimal conditions for the calcination of calcite are at the temperature of 898 ^{o}C, 1 atm pressure in a pure carbon dioxide atmosphere [13].

$$Calcination\ CaCO_3 \rightarrow CaO + CO_2 \tag{R2.1}$$
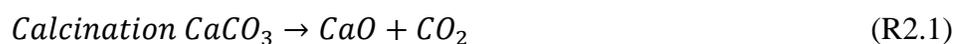
The calcination process is typically carried out using natural gas to ensure the high level of purity. The temperatures, flow of natural gas and fresh air have to be controlled properly to guarantee a high quality calcium oxide [12].

$$Hydration\ CaO + H_2O \rightarrow Ca(OH)_2 \qquad\qquad (R2.2)$$

$$Carbonation\ Ca(OH)_2 + CO_2 \rightarrow CaCO_3\ (\textbf{PCC}) + H_2O \qquad\qquad (R2.3)$$
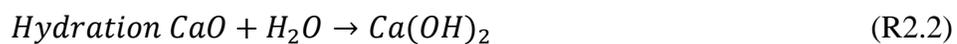
After calcination of limestone, the quicklime is crushed and screened and then reacted with water to produce so-called milk of lime (R2.2). Slaking of quicklime is followed by purifying the milk from the lime which is finally carbonated with the carbon dioxide obtained from the calcination process (R2.3) [14;15].
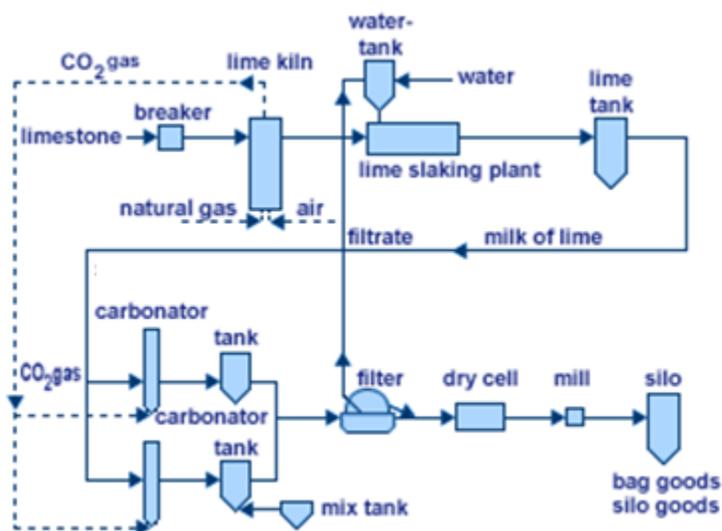
FIGURE 2.4 PCC production process [14].

2.2.3 Separation of PCC particles

The final stage in the PCC process is liquid solid separation by dewatering the suspension from reaction (R2.3). Dewatering is often performed by rotary vacuum filter, pressure filters or centrifuges. Particles in suspension are typically mono-dispersed with mean size 1-3 μm [16].

From dewatering a cake with 40-60 % moisture is obtained by filtering. The filtered water is recycled for producing lime slurry. The filter cake is dried in rotary, spray or flash driers at temperatures above 300 $^{\circ}$C. After drying, the PCC particles are thereafter pulverized into required mesh [14].

In some processes, filter cake is mixed with a dispersant and sent to a second stage centrifuge to produce smaller particles. The objective of the second-stage centrifuge in cooperation with the mill is to remove particles above size 25 μm and other unwanted particles. In this step, a fine-particle slurry with median size 5-8 um is dewatered to a high cake dryness of >70 % [16]. The fineness of the grain and the morphology of the PCC crystal can be modified during the process by controlling e.g. the temperature, concentration, pH and time.

2.3 Steelmaking

Steel is produced either in a basic oxygen furnace (BOF) or in an electric arc furnace (EAF), from iron, scrap and lime. In a BOF, pure oxygen is blown into the furnace to burn off the carbon from the molten iron, thus producing low-carbon steel, while in an EAF, electricity is used to melt scrap metal into steel [10].
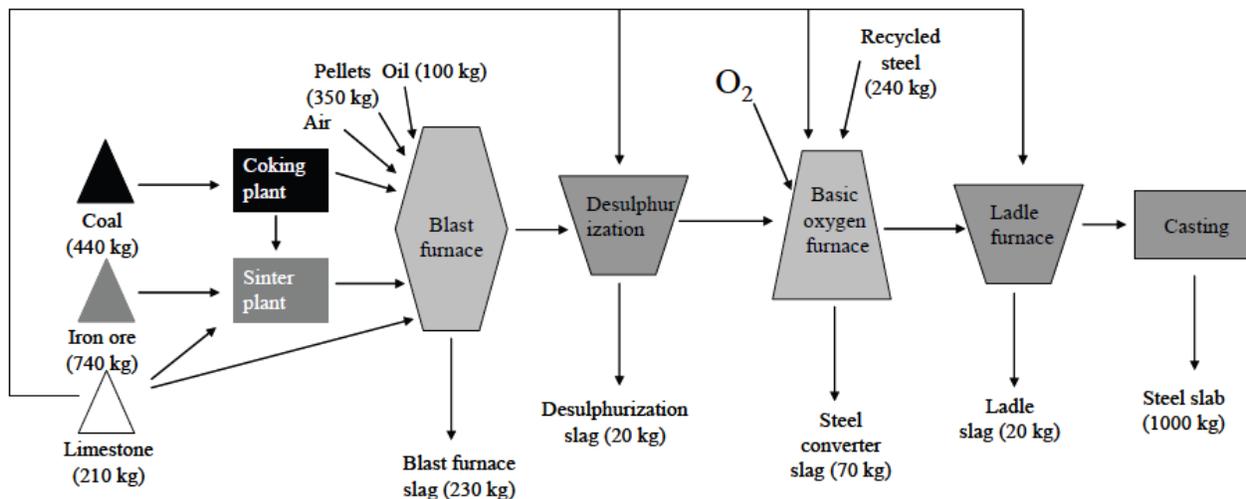
FIGURE 2.5 Simplified process model of the integrated route [10].

There are basically two different routes in a steelmaking process, being the integrated route and the electric arc furnace route, respectively. In the integrated route, in which BOF is used, steel is manufactured from iron ore, while in the electric arc furnace route recycled steel is fed and used as the raw material. A simplified process model of the integrated route is shown in figure 2.5.

There are four steel production plants located in Finland; Ruukki in Raahe, Outokumpu in Tornio, FNsteel in Koverhar, and Ovako in Imatra. The Raahe and the Koverhar plants use a BOF and the Imatra plant uses an EAF, while the Tornio plant uses Argon Oxygen Decarburization (AOD). In year 2011 the crude steel production in Finland was approximately 4.0 Mt, while the world output was about 1 490 Mt [17].

2.3.1 Basic oxygen furnace

The overall purpose of the BOF is to decarburize and remove the phosphorus in the steel and iron, and to raise the temperature of the liquid steel.

During the high temperatures of the liquid pig iron and the intense stirring in a BOF, an oxygen jet is introduced into the furnace. The oxygen jet contributes to a fast oxidation of the various components present in the liquid metal and a large resultant energy is released as heat. Silicon, manganese, iron and phosphorus form oxides, and in combination with the fluxes they create a liquid slag. Fluxes are minerals added early in the oxygen injection, to control sulfur and phosphorous and to control erosion of the furnace refractory lining. The oxidation reactions are expressed in (R2.4)-(R2.8):

$$C + \tfrac{1}{2}O_2 \rightarrow CO \qquad\qquad\qquad\qquad (R2.4)$$

$$Si + O_2 \rightarrow SiO_2 \qquad\qquad\qquad\qquad (R2.5)$$

$$Mn + \tfrac{1}{2}O_2 \rightarrow MnO \qquad\qquad\qquad\qquad (R2.6)$$

$$2P + \tfrac{5}{2}O_2 \rightarrow P_2O_5 \qquad\qquad\qquad\qquad (R2.7)$$

$$2Fe + \tfrac{3}{2}O_2 \rightarrow Fe_2O_3 \qquad\qquad\qquad\qquad (R2.8)$$

During the injection, limestone ($CaCO_3$) and dolomitic stone ($CaCO_3 \cdot MgCO_3$) are added in the BOF to control the chemistry and viscosity of the slag, but they can also serve as coolant. When limestone and dolomite are heated, an endothermic reaction occurs producing CaO, MgO and CO_2, see (R2.9)-(R2.10).

$$CaCO_3 + Heat \rightarrow CaO + CO_2 \qquad \text{(R2.9)}$$

$$MgCO_3 + Heat \rightarrow MgO + CO_2 \qquad \text{(R2.10)}$$

When the injection is finished, the slag floats up on top of the steel bath. Thus, by turning the furnace to the tap side, the steel can be tapped into a ladle. After tapping, the furnace is rotated to the other side to remove the slag into a slag pot. After the slag has been cooled, it is crushed and screened before being sold or otherwise disposed of.

The required quantities of hot metal, scrap, oxygen and fluxes vary according to their chemical composition and temperatures, and to the desired chemistry and temperature of the steel to be tapped.

2.3.2 Steelmaking slag

Iron and steelmaking slags are non-metallic by-products and consist primarily of calcium, magnesium, and aluminum silicates in various combinations. Slag is formed when limestone reacts with silicon dioxide and impurities of iron ore at high temperatures. The compositions vary with furnace type, composition of furnace charge, and individual furnace operating practice. The amount of slag produced is largely related to the chemistry of the raw material.

TABLE 2.1 XRF and TC results of slag from Ruukki in 2008, components over 0.1 % [5].

Component	CaO	Fe	SiO$_2$	Mn	MgO	Al$_2$O$_3$	V	Ti	P	Cr	S	C	Rest
%-wt	45.2	14.9	12.4	2.4	1.7	1.5	1.4	0.6	0.4	0.2	0.1	0.3	18.9

The slags obtained from steelmaking are usually named after the process they are generated by. Besides basic oxygen furnace slag, which is also called steel converter slag, and electric furnace

slag, desulphurization slag and ladle slag are also obtained during steelmaking, as can be seen in figure 2.5.

Steel slag can be used as an aggregate for concrete, asphaltic paving and road fill. Steel slags are also suitable as a source of iron and flux materials in blast furnaces and for improving soil quality in agriculture, while granulated blast furnace slag can be used as a partial replacement for, or additive to, Portland cement. Free oxide phases in steel slags can cause expansion, which restricts their use in construction applications that require very stable conditions [10].

The estimated world output of steel converter slag in year 2010 was approximately 120 to 180 Mt and of blast furnace slag approximately 230 to 270 Mt [18]. The four main steel plants in Finland produce approximately 1.6 Mt of different slags per year. In theory, if the slags produced in Finland could be utilized to manufacture precipitated calcium carbonate, about 1.1 Mt of calcium carbonate would be produced and approximately 0.5 Mt of CO_2 could be fixed annually [29].

2.4 Carbon dioxide capture and storage

2.4.1 Different stages of CCS

Carbon dioxide capture and storage (CCS) is an effective method for reducing carbon dioxide (CO_2) emissions from fossil fuel combustion. As mentioned above, the concept is mainly based on three different steps; removing the CO_2 from a gaseous emission (capturing), transporting the gas to a suitable location and storage in isolation from the atmosphere for a long period of time.

CCS systems would most likely be applied to large point sources of CO_2, such as large industrial processes or power plants. According to a special report on carbon dioxide capture and storage by the Intergovernmental Panel on Climate Change (IPCC), CO_2 emissions from a power plant equipped with a CCS system could be lowered by about 80-90 % compared to a plant without CCS [3].

Capture technologies separate carbon dioxide from gases produced in electricity generation, this can currently be done in at least three different ways; pre-combustion capture, post-combustion

capture and oxyfuel combustion. A pre-combustion includes converting solid, liquid or gaseous fuel into a mixture of hydrogen, carbon dioxide and other gases by a number of processes, while post-combustion involves absorbing the CO_2 in a solvent from the exhaust. Oxyfuel combustion systems use oxygen mixed with CO_2 instead of air for combustion, resulting into a flue-gas consisting mainly of CO_2 and H_2O [3].

For transporting different gases, pipelines are today commonly used and are considered the best alternative for CO_2 for longer distances. The CO_2 is usually pre-treated by compressing it above 8 MPa to avoid two-phase regimes in order to make the transport easier and less costly. Similar technology has been utilized for transporting natural gas and oil. Another alternative for transport is by using ships or road tankers. By using mobile units, the gas can be transported as a liquid at temperatures lower than ambient and pressures >4.5 bar.

Once the CO_2 has been transported, it can be stored several hundred meters below the earth's surface, which is the "conventional" CCS method. Typical storage places are former gas and oil fields or deep saline formations. The storage places have to be carefully selected to ensure that the CO_2 is completely and safely trapped. In Finland, however, underground storage capacity for CO_2 does not exist [19].

CCS technology is still in the demonstration phase and is considered not risk free. Besides a climate change effect of CO_2 returning to the atmosphere, leakages can also have a dangerous impact on the ecosystem and human health.

2.4.2 Mineral carbonation

A different route for CCS is mineral carbonation. Mineral carbonation involves reacting carbon dioxide with earth metal oxides, such as magnesium oxide (MgO) and calcium oxide (CaO). These metal oxides occur naturally in silicate rocks, like serpentine and olivine, but also in small yet significant quantities of industrial residue, such as steel slag. The main end product of mineral carbonation is magnesium carbonate ($MgCO_3$) or limestone ($CaCO_3$).

The risk related to mineral carbonation compared to the other CCS-techniques is considered very low. CO_2 in carbonates is more stable and therefore no long-term monitoring of CO_2-leakage is required. However, the utilization of the end product is largely an open question. The storage and use alternatives all have limited space and longer transportation of the carbonates implies extra costs. To fix about one ton of CO_2 is estimated to require about 1.6 to 3.7 tons of rock, and produce 2.6 to 4.7 tons of material to be disposed per ton of CO_2 stored as carbonates [3]. On the other hand, CCS by mineral carbonation can be used to produce the material for land reclamation; this is currently in development in Singapore, in cooperation with Åbo Akademi University.
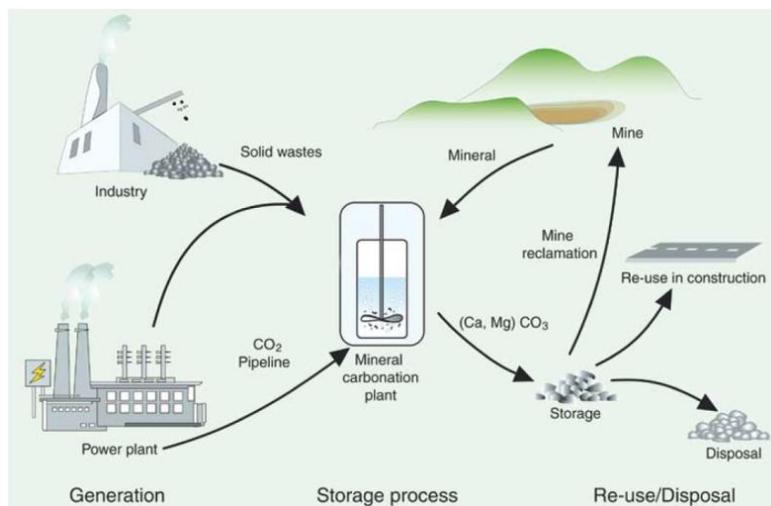
FIGURE 2.6 Different process steps for mineral carbonation [3].

Mineral carbonation also occurs naturally, known as weathering. Natural weathering occurs spontaneously when minerals, such as limestone and chalk, is exposed atmospheric CO_2 and rain. However, natural weathering is a very slow process and is not suitable for industrial purposes unless the reaction is accelerated e.g. by pre-treatment of the mineral.

Current research on mineral carbonation therefore focuses on accelerating the process of the carbonation in an energy-efficient way and using the end product for commercial use. This is also the aim of the task within the Cleen CCSP-project, as stated in the introduction. By using ammonium salts for dissolving the calcium from steel converter slag and thereafter carbonating the solvent, low value slag is reused and does not have to be landfilled, carbon dioxide emissions are reduced, and the end product has a demand in the commercial markets.

3 LABORATORY-SCALE TWO-STAGE CARBONATION EXPERIMENTS WITH AMMONIUM SALTS

3.1 Experiment description

Two laboratory-scale extraction-carbonation experiments with ammonium salt solutions were performed prior to a larger scale experiment. In the first experiment, referred here as experiment 1, 1 mol/L ammonium nitrate (NH_4NO_3) solution was utilized with steel converter slag from Ruukki Raahe Works. The slag, which was received in 2008, was sieved to a 125-250 μm particle size fraction. The process was similar to previous work of Mattila and Grigaliūnaitė; it was considered as semi-continuous since the end product was not removed continuously [6].

During the Cleen CCSP-project in 2011, Ruukki produced a slag in their lab, as part of work on making modifications to their steelmaking process. This changed the slag composition; it has been reported to have a higher vanadium (V) concentration and less free calcium [20]. To determine the extraction of Ca from the 2011 slag, a second experiment was performed. The second experiment, referred here as experiment 2, was a batch process with 1 M ammonium chloride (NH_4Cl)-solution. A batch of the 2011 slag was received in 2011 from Ruukki's laboratory and, similar to experiment 1, sieved to a 125-250 μm particle size fraction. Both experiments were performed under ambient conditions (~20 ^{o}C, 1 bar).

3.2 Experiment setup

The process setup of experiment 1 is presented in figure 3.1. As shown in the figure, the process consisted of four filters, four pumps, a motor stirrer, a pH adjustment unit and a CO_2 removal unit. The different volume flow rates are expressed in table 3.1.

During experiment 1, the motor stirrer continuously mixed the suspension at 170 rpm. Carbon dioxide (99.9996%) was fed at 1 L/min into the carbonation reactor, while nitrogen (99.9999%, Oy Aga Ab) was injected at 0.5 L/min into the CO_2 removal unit to remove unreacted carbon dioxide.
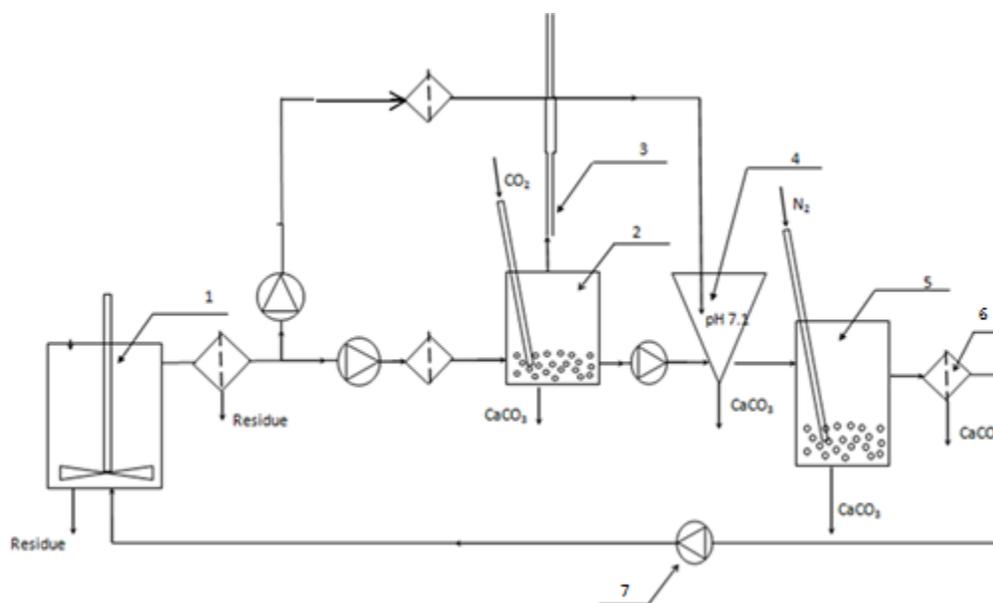
FIGURE 3.1 Process scheme for experiment 1; 1 - Extraction; 2 - Carbonation; 3 - water lock; 4 - pH adjustment unit; 5 - CO_2 removal unit; 6 - filtration; 7 – pump [6].

Temperature and pH in the extraction and carbonation reactor were measured with an ion selective electrode (ISE, Nico2000) for pH and its temperature probe during experiment 1. An additional pH meter (Precisa pH900) was positioned to measure the pH-value in the pH adjustment unit.

TABLE 3.1 Volumes, volume flows and residence times in experiment 1.

Unit	Volume (ml)	Volume flow (ml/min)	Residence time (min)
Extraction	500	20	25
Carbonation	300	15	20
pH adjustment	500	20	25
CO_2 removal	500	20	25
Filter equipment	600	20	

Experiment 2 consisted of a single vessel acting as reactor as the two stages, extraction and carbonation, were performed separately. The reactor was equipped with a motor stirrer, which continuously mixed the suspension at 170 rpm during the extraction. For carbonation, the motor stirrer was removed and replaced by a CO_2-gas distributor, which injected CO_2 at approximately 1 L/min. The gas bubble flow gave mixing instead of a mixer. For pH and temperature

measurements an ISE was utilized in experiment 2. All measurement devices were calibrated before the experiments.

3.3 Experiment performance

Before initiating experiment 1, the system was filled with a 1 mol/L NH_4NO_3-solution. A total volume of 2500 ml solution was used and the volumes in different steps are shown in table 3.1.

50 g slag was initially fed to extraction reactor, giving a solid-to-liquid ratio of 100 g/L, and thereafter 5 g slag was added every 15 minutes. The total duration of experiment 1 was 255 minutes and a total of 110 g slag was utilized.

During experiment 1, suspension consisting of fine particles was removed from the extractor through an overflow. This suspension was filtered to prevent impurities from entering the carbonation stages. Approximately 75% of the suspension was pumped at 15 ml/min to the first carbonation step, while 25% was pumped at 5 ml/min to the second precipitation step, the pH adjustment unit (see Figure 3.1). The second precipitation step is used for increasing the pH of the once carbonated solution to approximately 7 for additional precipitation of calcium carbonate. Before recycling the suspension back to the extractor, the liquid was directed through a final vessel for removal of unreacted CO_2.

Samples were taken of precipitated calcium carbonate at different stages of the experiments. They were filtered, dried overnight at 100 ^{o}C, weighed and analysed with a scanning electron microscope (SEM/EDX). The suspensions were analysed with an ISE to determine the concentration of dissolved calcium.
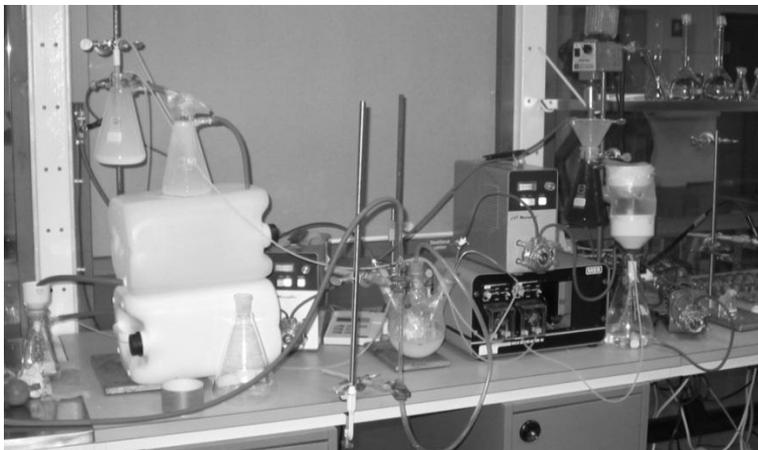
FIGURE 3.2 Process equipment of experiment 1.

In experiment 2, 50 g of the new slag was fed in the reactor for extraction, giving a solid-to-liquid ratio of 100 g/L. Samples of 10 ml were taken at 20 minutes, 40 minutes and 60 minutes during extraction to determine the solubility of the new slag. Duration of extraction was one hour.

Carbonation was performed after extraction. The duration of carbonation was one hour and 10 ml samples of suspension were taken at 15, 30, 45 and 60 minutes.

3.4 Results

3.4.1 Produced precipitated calcium carbonate

After experiment 1, the end product (PCC) was filtered, dried and weighed. The samples were taken from the carbonator, pH adjustment unit and CO_2 removal unit, respectively, and they together weighed 23.25 g. Approximately 35% of the end product was found in carbonator, while 60% and 5% were found in pH adjustment unit and CO_2 removal unit, respectively.
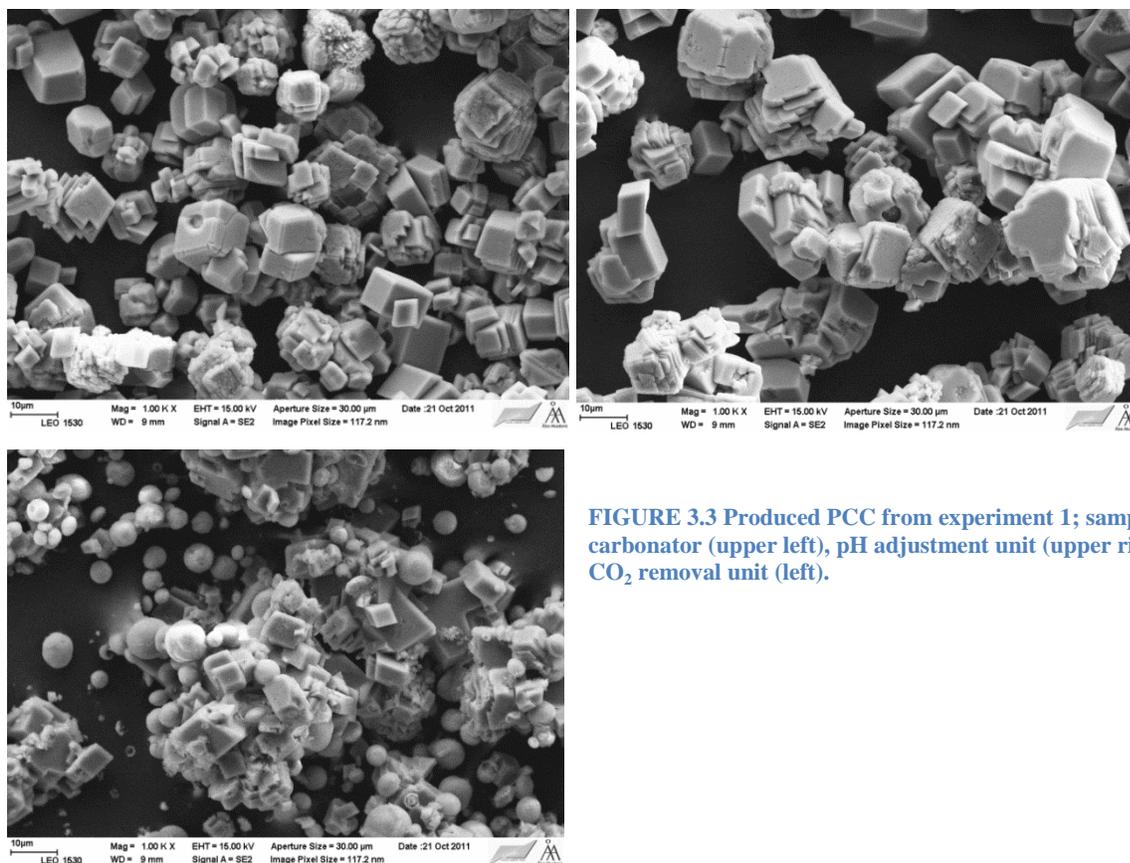
FIGURE 3.3 Produced PCC from experiment 1; sample from carbonator (upper left), pH adjustment unit (upper right) and CO_2 removal unit (left).

The end product was analysed with SEM/EDX to determine its crystalline form and chemical composition (figure 3.3). Samples from the carbonator and pH adjustment unit seemed to contain rhombic calcite, while in the CO_2 removal unit some spheric vaterite was found. Although a small percent of iron (0.61% Fe) was found in the sample from the CO_2 removal unit the end product was considered very pure, as no impurities at the detection limit were found in first two steps of the process in experiment 1. In experiment 2 approximately 2.67 g PCC was found in the carbonator.

3.4.2 Extraction

Concentrations of calcium in the suspensions were measured with an ISE. One sample was taken from both the extractor and carbonator after experiment 1, while three samples were taken during extraction of experiment 2 and four samples during carbonation, respectively.

TABLE 3.2 Ca²⁺ concentrations in carbonation (upper table) and extraction (lower table).

Carbonation time (min)	15	30	45	60	255
Experiment 1, Ca²⁺ (mol/L)					0.0038
Experiment 2, Ca²⁺ (mol/L)	0.00623	0.00605	0.00671	0.00659	

Extraction time (min)	20	40	60	255
Experiment 1, Ca²⁺ (mol/L)				0.0175
Experiment 2, Ca²⁺ (mol/L)	0.066	0.0805	0.0926	

Based on Ca^{2+} concentrations in the reactors, the conversion efficiency of carbonation can be determined by equation (E3.1):

$$\eta_{conversion}(\%) = \frac{c_{extractor} - c_{carbonator}}{c_{extractor}} \qquad (E3.1)$$

The conversion efficiency in experiment 1 was approximately 78.3%, while the efficiency in experiment 2 was approximately 92.9%. The concentrations from experiment 2 were taken at 60 minutes, after the processes were terminated.

As approximately 87 g residual slag was obtained from a total of 110 g slag in experiment 1, the extraction efficiency resulted to ~21%. In experiment 2, on the other hand, 46.27 g residue was obtained from 50 g slag, giving an extraction efficiency of only 7.5%.

3.4.3 Measured pH and temperature

Temperature and pH changes were measured during both experiments. Comparing pH values at extraction shown in figure 3.4, one can see the similarity from both experiments. The duration of experiment 2 was only 60 minutes but the pH in the extractor was nonetheless almost identical to that of experiment 1. The fact that the pH decreased with time in experiment 1 indicates that some minor precipitation of carbonates occurred in the extractor.

Any precipitation did obviously not occur during extraction in experiment 2 because no CO_2 was present in the process.
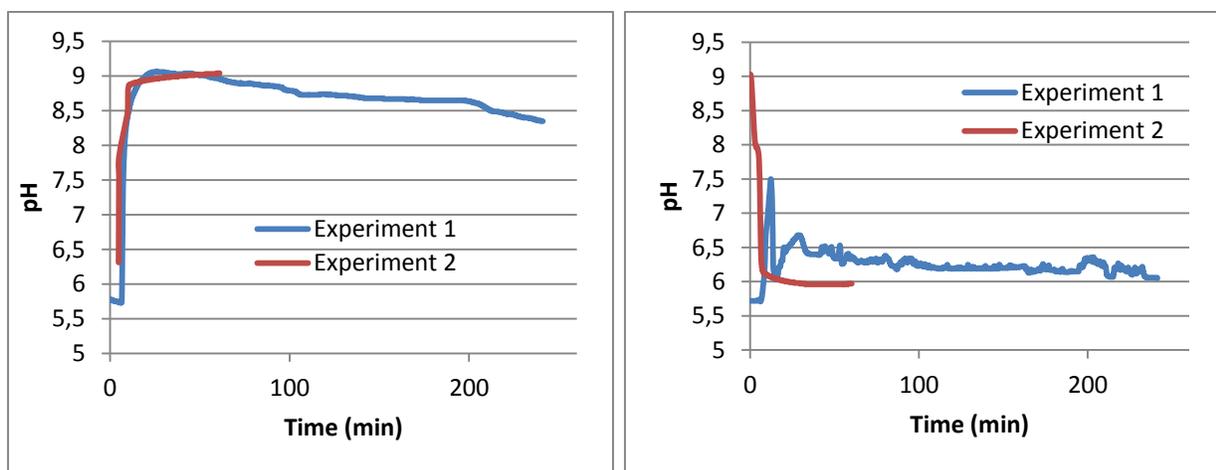
FIGURE 3.4 Measured pH changes during extraction (left) and carbonation (right) of experiment 1 and 2.

During carbonation in experiment 2, the pH value decreased rapidly during the first 10 minutes and was thereafter stable throughout the process. The pH value during carbonation in experiment 1 was quite inconsistent in the beginning but stabilized after about 30 minutes.

As mentioned earlier, extraction and carbonation in experiment 2 were performed separately but their temperature changes are both presented in figure 3.5. Only one temperature probe was used during experiment 1; it was positioned in the extractor and should not be compared with carbonation in experiment 2.

In the beginning of the extraction in both experiments, an increase in temperature was noted as a result of Ca dissolution heat. Although the increase of temperatures slowed down with time, extraction temperature in experiment 1 was approximately 1 $^{\circ}$C higher than in experiment 2. The temperature difference between experiment 1 and 2 for the extraction stage may depend on the different slag and solvent used in the experiments.
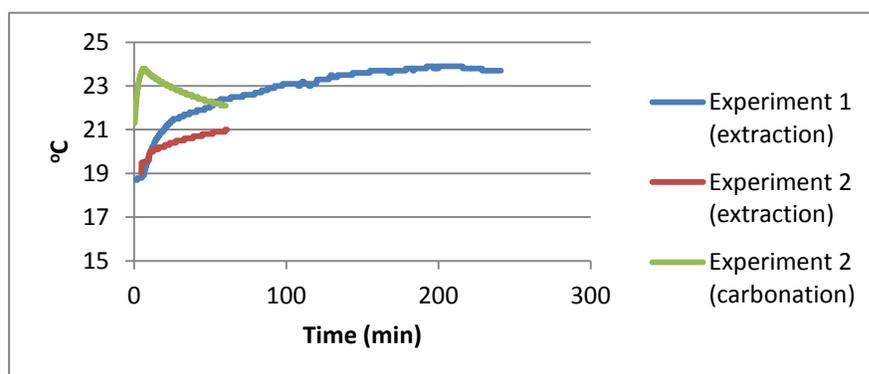
FIGURE 3.5 Temperature changes during experiments.

The fact that carbonation is an exothermic reaction is also shown in Figure 3.5. Temperature increased in a rapid pace during the first 7 minutes of carbonation in experiment 2, reaching its peak at 23.8 ^{o}C. The temperature started to decrease after 7 minutes, which indicated that the carbonation reaction was slowing down.

4   CONTINUOUS SEPARATION BY GRAVITY SEDIMENTATION USING INCLINED SETTLER

4.1 Experiment description

As part of scaling up the extraction-carbonation process, continuous particle separation from a liquid using an inclined settler was investigated. Several experiments with steel converter slag from Ruukki Raahe Works and Ballotini glass beads as particles were performed separately to determine the feasibility of the separation technique. Ballotini glass beads, with a density of approximately 2634 kg/m^3, were sieved to a size fraction of 125-212 μm, while the measured density of steel converter slag particles (as residue) was 2604 kg/m^3 and sieved to size fractions of <125 μm and 63-125 μm.

In these experiments, water at ambient temperature (~20 ^{o}C) was used as liquid. Water density and viscosity were not measured before the experiments, but according to literature the properties are approximately 998.2 kg/m^3 and 0.001 Pa·s, respectively [21]. Ammonium salt solutions, which are used in extraction-carbonation experiments, were not utilized here because water has somewhat similar properties as ammonium salt solutions (1015 kg/m^3, 0.00098 Pa·s [21;22]) and extraction properties of the particles were not studied in these experiments.

4.2 Experiment setup

Acting as reactor, a cylindrical perspex vessel with a total volume of 30 L and 26 cm diameter, was equipped with a motor stirrer. The motor stirrer continuously mixed the suspension at approximately 170 rpm during all experiments. Suspension was pumped from the reactor with a peristaltic pump (Millipore XX 80 002 30) into the separator.

The inclined settler, a 110 cm long circular perspex tube with 10 cm diameter was in addition to the suspension feed flow also equipped with an underflow outlet at the bottom and an overflow at the top. Both underflow and overflow streams were continuously recycled back to the reactor.

4.3 Performance of experiments

Three experiments were performed with slag particles and eighteen experiments with glass beads with the separator inclined in four different angles (0°, 10°, 30° and 45°, respectively, with respect to horizontal). Volume flows from the reactor in experiments with slag particles were adjusted at approximately 40 L/h, while experiments with glass beads the volume flow were set at 40-65 L/h.
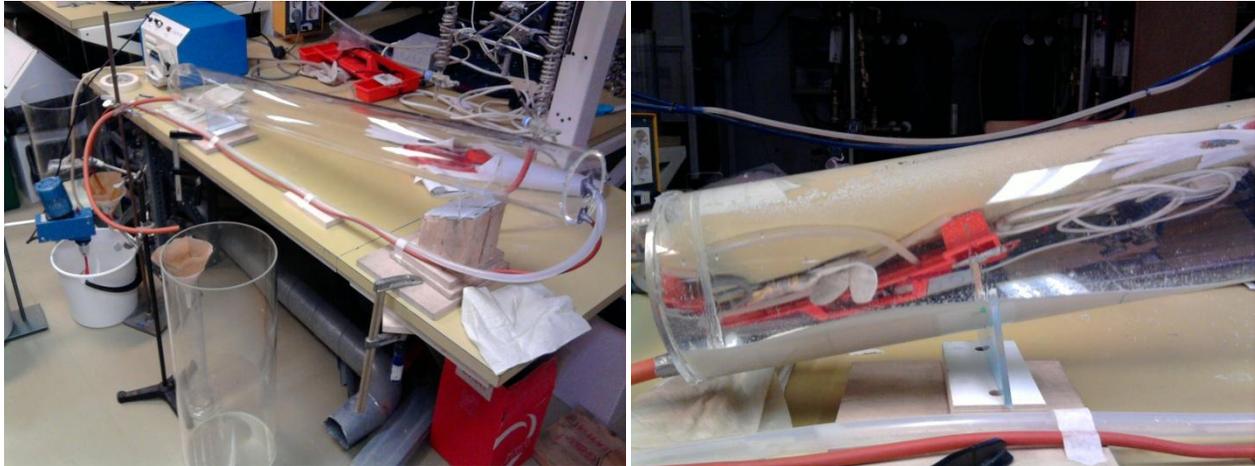
FIGURE 4.1 Process setup for experiment with 10° inclined settler.

Before engaging the process, the separator was filled with liquid up to the overflow orifice. The overflow orifice was located at the top of separator, except for experiments at 0° inclination; the orifice was in that case positioned in the center of the other end. Particles were thereafter added into the reactor and mixed properly. The initial particle volume concentration in reactor was in all experiments about 100 g/L.

Separation and removal of particles was performed through the underflow of the settler. The underflow volume rate was adjusted to approximately 7 L/h, but it was very irregular due to a high particle volume concentration which occasionally blocked the stream. A pump or a larger diameter for the underflow would be preferable in future work.

Movement of the suspension and the settling of particles were observed visually. The partial dissolution of slag particles even in pure water resulted into a very turbid suspension which

caused some problems to determine where the particles had settled. This issue did not occur with glass beads because the particles naturally do not dissolve in water.

Samples from the overflow in experiments with slag particles were taken during the last 10 minutes of a test to determine overflow particle concentration. The samples were filtered, dried overnight at 100 ^{o}C, and weighed. This was not performed for glass beads because no particles reached the overflow stream.

4.4 Results

4.4.1 Measured particle volume concentrations

Initial particle volume concentration in the reactor was approximately 100 g/L for all tests. Basically, once the particles have entered the separator, they will settle onto the inclined wall and be removed through the underflow. In almost all experiments, some particles remained on the wall, changing the particle volume concentration in the reactor to approximately 90 g/L. By assuming good mixing of particles in the reactor, particle volume concentration is considered identical for both the reactor and the feed flow.

FIGURE 4.2 Inclined settler at 30° angle (right) and reactor samples from the experiment (left).

The particle volume concentrations from underflow were not measured during the experiments due to its irregular flow. It is, however, possible to calculate the concentrations from a mass balance equation (E4.1) assuming a steady flow.

$$Q_{in}c_{in} = Q_u c_u + Q_o c_o \qquad \text{(E4.1)}$$

where the subscripts in, u and o are inflow, underflow and overflow, respectively. Experiments with glass beads were not analysed with equation (E4.1) since no particles reached the overflow.

TABLE 4.1 Measured and calculated concentrations for experiments with BOF slag.

Material	Fraction (µm)	Angle (°)	Inflow Q_{in} (L/h)	Underflow Q_u (L/h)	Overflow Q_o (L/h)	c_{in} (g/L)	c_o (g/L)	c_u (g/L)
BOF slag	< 125	10	40	7	33	90	0.129	513.68
	63-125	30	40	7	33	90	0.129	513.68
	63-125	45	40	7	33	90	0.127	513.69

Overflow was directed to a second vessel during the last 10 minutes of the experiments to determine particle volume concentrations of the overflow. The samples, which contained approximately 5.5 L suspension each, were filtered, dried and weighed. Measured concentrations are shown in table 4.1, along with the calculated concentrations from equation (E4.1).

As one can see, particle concentrations from different inclinations do not differ much from each other. Separation of the solid particles from the liquid was effective; the larger particles settled in the separator while mostly calcium from the slag dissolved in the water. Silicates formed a dispersion of small, micrometer scale particles in liquid (based on SEM/EDX results). As long as the sediment of slag particles does not reach the overflow stream, particle volume concentration will remain similar in the reactor and the separator.

4.4.2 Particle settling

Particle settling and suspension movement were observed visually. Separation of particles started when suspension was pumped into the separator and it was continuous during all experiments. Ballotini glass beads settled quite fast due to their large sizes and mass, while slag particle settled at a much slower rate. As the particles settle at different velocities, they form sediment along the inclined settler. The sediment lengths of glass beads and slag particles at different volume flows are plotted in figure 4.3.
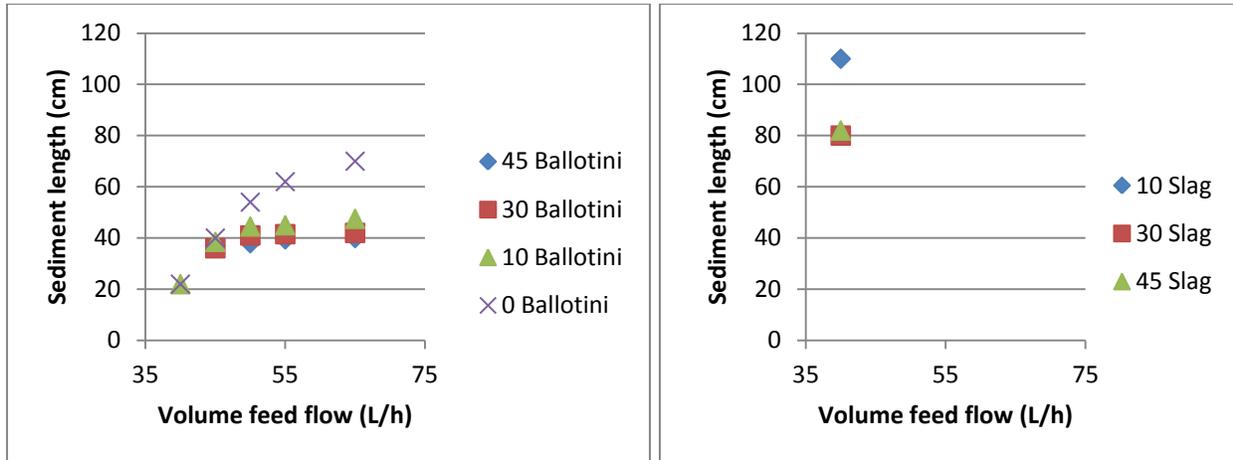
FIGURE 4.3 Sediment lengths of glass beads (left) and slag particles (right) in different volume flows and settler inclinations.

Sediment ranges of slag particles were observed to be much longer than glass beads, shown in figure 4.3. Slag particles with size fraction 63-125 μm in a 30° and 45° inclined settler formed an 82 cm and 80 cm, respectively, range of sediment in the separator. Although slag particles with size fraction <125 μm reached the overflow orifice, no notable changes in the overflow particle volume concentration were detected. Experiments with steel converter slag were only performed with feed flows of 40 L/h.

Experiments with glass beads were performed in 40-65 L/h feed flows. The length of particle sediment followed a similar pattern in various inclinations, with the exception in experiments with 0° inclination. In experiments with 0° inclination the sediment range was about 70 cm at the highest volume flow tested (65 L/h), while the sediment range in experiments with inclinations of 10°-45° was around 40-48 cm for 65 L/h.

These experimental results can be compared with theory of particle gravity settling. Particle settling rate in fluids is determined by Stokes' law on terminal settling velocity [23].

$$U_t = \frac{k \cdot \Delta\rho \cdot g \cdot x^2}{18 \cdot \mu} \qquad (E4.2)$$

where $\Delta\rho$ is the density difference between the solid and the liquid, g is gravitational acceleration (~9.81 m/s^2), x is the single particle size and μ is the liquid viscosity, while k is a correction factor which is used for suspensions with >0.1 % particle volume concentration. In dilute suspensions the particles interact with each other and hinder each other's fall (called hindered settling) [23]. The most frequently used correction factor is developed by Richardson and Zaki,

$$k = (1 - c)^n \tag{E4.3}$$

where n is a variable constant that depends on the particle Reynolds number (and also on vessel diameter [D]).

TABLE 4.2 Variable constant for Richardson and Zaki's correction factor at different Reynolds number [23].

Particle Reynolds number	n for small tubes	n for large tubes
< 0.2	4.65 + 19.5 x/D	4.65
0.2 < Re < 1	(4.35 + 17.5 x/D) Re$^{-0.03}$	4.35 Re$^{-0.03}$
1 < Re < 200	(4.45 + 18 x/D) Re$^{-0.1}$	4.45 Re$^{-0.1}$
200 < Re < 500	4.45 Re$^{-0.1}$	4.45 Re$^{-0.1}$
Re >500	2.39	2.39

The particle Reynolds number is calculated as

$$Re_p = \frac{x U_p \rho}{\mu} \tag{E4.4}$$

where U_p is particle velocity inside the separator

$$U_p = \frac{U_t}{k} - U_f \cdot \sin\theta \tag{E4.5}$$

$$U_f = \frac{4 Q_o}{\pi D^2} \tag{E4.6}$$

If particle Reynolds number exceeds 0.2 the degree of turbulence becomes more significant and affects the drag force [23].

In addition, separation efficiency increases with separator being inclined. Thus, this needs to be taken into account. A characteristic clarification rate, based on the PNK-theory (Ponder (1925), Nakamura and Kuroda (1937)), states that the volumetric rate of production of clarified fluid is equal to the vertical settling velocity of the particles multiplied by the horizontal projected area of the upward facing surface of the channel onto which the particle may settle, assuming that all of the particles have the same settling velocity [24]. The equation is expressed as

$$S = U_t D (L \cdot \sin \theta + D \cdot \cos \theta) \tag{E4.7}$$

where S is the characteristic clarification rate (m^3/s), θ is the angle of the inclination with respect to the vertical and L is the length of the separator.

By using equation (E4.5), the cutoff sedimentation velocity can be determined from different overflow volume rates, being $Q_o = S(U_t)$. If the terminal settling velocity for a given particle is lower than the cutoff sedimentation velocity, the given particle will reach the overflow. Also, by use of equation (E4.2) combined with (E4.5), the size of the largest particles (cutoff particle size) to reach the overflow can be calculated. The cutoff particle size, also called particle cut size, signifies the size of particles that have a 50 % chance to reach the overflow.

The terminal settling velocity of glass beads, using $\overline{d}_g = 163$ μm and $k = 0.64$, is $U_{t,g} \approx 15$ mm/s. For comparison, table 4.3 presents the cutoff particle sizes and cutoff sedimentation velocity for glass beads at different overflows used in the experiments. Table 4.3 is calculated with equation (E4.2) combined with (E4.5).

As one can see in the table, the terminal settling velocity ($U_{t,g}$) for glass beads is much higher than the cutoff sedimentation velocities. For glass beads to reach the overflow orifice, their terminal settling velocities need to be equal as or lower than the cutoff sedimentation velocities presented in table 4.3. Also cutoff particle sizes show that glass beads were too large to reach the overflow.

TABLE 4.3 Cutoff sedimentation velocities and cutoff particle sizes at different overflows and inclinations for Ballotini glass beads.

Inflow (L/h)		40	45	50	55	65
Overflow (L/h)		33	38	43	48	58
Material	Inclination	Cutoff sedimentation velocity (mm/s)				
Glass beads	0º	0.092	0.106	0.119	0.133	0.161
	10º	0.078	0.090	0.102	0.113	0.137
	30º	0.065	0.075	0.084	0.094	0.114
	45º	0.062	0.071	0.080	0.090	0.108
		Cutoff particle size (µm)				
	0º	12.68	13.60	14.47	15.29	16.80
	10º	11.69	12.54	13.34	14.10	15.50
	30º	10.65	11.43	12.16	12.85	14.12
	45º	10.40	11.16	11.87	12.55	13.79

The terminal settling velocity of slag particles with size fraction 63-125 µm, by using average size $\overline{d}_{s1}$ = 89 µm and k = 0.64, is $U_{t,s1}$ = 4.4 mm/s. The cutoff sedimentation velocity and cutoff particle size for slag particles with separator in 30º to 45º inclinations were about 0.06 mm/s and 10.5 µm, respectively. For slag particles with size fraction <125 µm ($\overline{d}_{s2}$ = 11.2 µm, k = 0.64) the terminal velocity is $U_{t,s2}$ = 0.07 mm/s. As the cutoff particle size is 11.8 µm for slag particles with separator in 10º inclination, a small fraction of particles reached the overflow orifice.

4.4.3 Separation efficiency

As for all separation methods, the efficiency is calculated based on the difference of concentration in and the concentration out,

$$\eta = 1 - \frac{c_o}{c_{in}} \cdot 100\% \qquad (E4.8)$$

where η is the separation efficiency. Separation efficiency of all experiments with glass beads is obviously 100 % as no particles reached the overflow. Separation efficiencies of experiments with slag particles are shown in table 4.4.

TABLE 4.4 Separation efficiency of slag particles and glass beads.

Material	Size fraction (μm)	Inclination	c_{in} (g/L)	c_o (g/L)	η
BOF slag	<125	10°	90	0.129	99.86 %
	63-125	30°	90	0.129	99.86 %
	63-125	45°	90	0.127	99.86 %
Glass beads	125-212	0°-45°	90	0	100 %

Although the slag particle size fraction was <125 μm at a 10° inclination, the efficiency did not differ from particles with size fraction 63-125 μm even at larger inclinations. Particle volume concentration of the suspension at the top of the separator was similar in all experiments with slag particles. This mainly indicates that only when the sediment of settled particles reaches the overflow, the concentration in the overflow will increase and separation efficiency decreases.

5   LARGER SCALE EXTRACTION AND CARBONATION EXPERIMENT WITH CONTINUOUS SEPARATION USING AN INCLINED SETTLER

5.1 Experimental description

Two experiments, extraction and carbonation, respectively, were performed separately, to represent a realistic view of the continuous process. 1 M ammonium chloride (NH_4Cl) was utilized as solvent to dissolve calcium from steel slag.

The slag was received in year 2011 from Ruukki Raahe Works and had an approximate density of 2720 kg/m^3 (measured after extraction). It should be noted that the slag was a laboratory test slag and not from an actual full scale process. This is the same laboratory slag used in experiment 2 in chapter 3. Although the size fraction of slag particles in the previous experiments has been 125-250 μm, it was now widened to cover 63-425 μm due to the limited amount of slag available (~5 kg).

5.2 Experimental setup

Acting as reactor, a perspex vessel (diameter 26 cm, height 60 cm) with a total volume of 30 L was chosen and equipped with a motor stirrer. The stirrer continuously mixed the solvent at ~170 rpm during both experiments (extraction and carbonation). An ELIT ISE and temperature probe were utilized for pH and temperature measurements in the reactor. In addition, a Precisa pH900 was positioned to measure the pH of the recycled suspension. The pH meters were calibrated right before the experiments.

Separation of particles from the solvent was performed using sedimentation by a 1.1 m long perspex tube (diameter 10 cm) inclined settler with a total volume of 8.64 L (see figure 5.1). The separation tube was also used in the above mentioned experiments (chapter 4). The settling tube was inclined 45$^{\text{o}}$ with respect to horizontal, and equipped with a feed flow, underflow and overflow. The feed flow orifice was positioned at the top of the base and the underflow was positioned below the feed flow orifice, while the overflow orifice was situated at the top end. The

purpose of these flows is for the particles to settle at the inclined wall and roll down to the base and be removed through the underflow, while clear suspension exits through the overflow.

A peristaltic (Millipore XX 80 002 30) pump was used to transport the solvent through a tube into the inclined settler. Both the underflow and overflow were also equipped with a silicon tube to recycle the slag and suspension back to the reactor.

FIGURE 5.1 Process setup for extraction experiment.

5.3 Experiment performance

The experiments were performed at ambient temperature (~20 ^{o}C), which is advantageous for economic reasons. Heating up the process would result in higher energy costs and vaporization of ammonia at a faster rate, but earlier work has shown that ambient pressure and temperature give good performance.

5.3.1 Extraction experiment

A total volume of 20 L 1 mol/L NH_4Cl-solution was used for extraction, of which approximately 8.25 L was pumped into the settler before mixing the slag into the solvent solution. Steel slag was added to the remaining 11.75 L in the reactor, giving an initial particle volume concentration of 100 g/L. The slag was initially mixed in the solvent for about 30 minutes before engaging the separation process.

The suspension was thereafter pumped into the inclined settling tube with an approximate volume flow of 40 L/h, and directed back through the underflow and overflow. The underflow rate was adjusted to approximately 7 L/h, but was at times irregular due to the high concentration of the sediment which occasionally blocked the stream.

After 75 minutes the process was considered to be in a steady-state and the overflow was directed into a second vessel, from which it was possible to determine the particle volume concentration in the overflow. The total time length of the experiment was approximately 100 minutes. The calcium-rich solution obtained was saved for the carbonation experiment.

5.3.2 Carbonation experiment

The carbonation step was equipped and performed similar to the extraction step, except for the CO_2 gas feed and some minor changes in the separation stage. Different from the extraction experiment, the fluid was not pumped into the inclined settler before initiating the carbonation experiment; dissolved Ca was first carbonated with CO_2 gas and the solution was thereafter pumped into the settling tube.

The solution containing the calcium from the extraction experiment was filtered from impurities before the carbonation. Some volume losses were observed, as the total volume of the solvent for the carbonation experiment was approximately 18.8 L.

The solvent was mixed continuously during the experiment with a motor stirrer at approximately 170 rpm. The flow rate of the CO_2-gas was approximately 2.2 L/min and the carbonation was terminated after 60 minutes. After approximately 40 minutes, the solvent was pumped into the

inclined settler, and once filled (~8.25 L), at 50 minutes, the feed flow was adjusted to approximately 40 L/h.

Samples were taken at 65 minutes and 80 minutes, respectively, to determine the PCC particle crystal shape and concentration in the different flows. The total duration of the carbonation experiment was about 85 minutes.

FIGURE 5.2 The separator (left) and reactor (right) in carbonation experiment.

5.4 Results

5.4.1 Precipitation of calcium carbonate

The carbonate precipitation reactions can be summarised as shown in (R5.1)-(R5.2). Before the calcium carbonate precipitates, gaseous carbon dioxide must be absorbed in the process liquid. After the carbonation experiment, the liquid solvent containing the PCC particles was vacuum filtered using Whatman filters. The filtrates were dried overnight at ~100 ^{o}C and weighed.

$$2NH_4OH(aq) + CO_2(g) \leftrightarrow (NH_4)_2CO_3(aq) + H_2O(l),$$
$$\Delta H = -110kJ/mol \quad (R5.1)$$

$$(NH_4)_2CO_3(aq) + CaCl_2(aq) \leftrightarrow CaCO_3(s) + 2NH_4Cl(aq),$$
$$\Delta H = 15 - 20kJ/mol \ (R5.2)$$
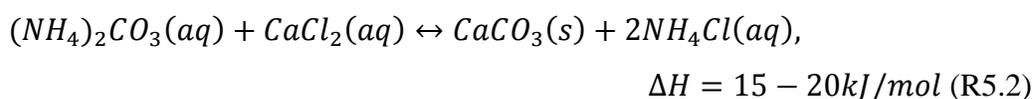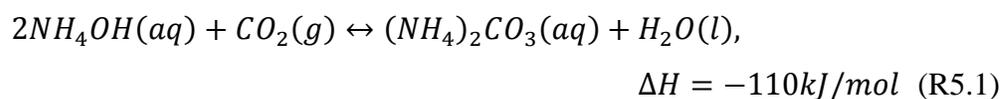
The total amount of PCC produced was approximately 85.37 g. From the reactor 29.92 g PCC was found, while approximately 37.26 g PCC had settled in the settling tube. Also in the samples, taken from the underflow and the overflow from the settler, approximately 18.06 g and 0.13 g PCC was obtained, respectively.
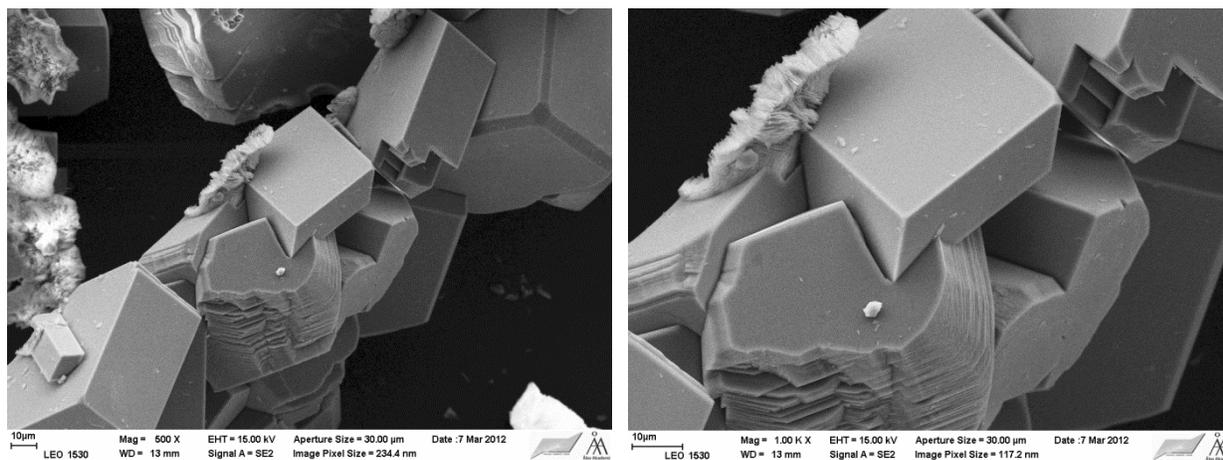
FIGURE 5.3 SEM/EDX pictures of samples from the overflow at 80 minutes.

Analysing the samples with SEM/EDX reveals that mostly rhombohedral calcite was produced but also some vaterite occurred. The PCC particles were considered relatively pure, although some chloride (0.14%-0.30%) from the solution was found in every sample. The sizes of the particles were about 10-30 μm – see figure 5.3 (which also shows the irregular shaped chloride).

5.4.2 Measured Ca^{2+} concentrations

Concentrations of calcium were measured from samples taken during and after the extraction and carbonation. In extraction, two samples were taken during the experiment, at 60 minutes and at 70 minutes, from the overflow, and two samples after experiment was terminated, one from the reactor and one from the second vessel. Approximately 10 ml of each sample was enough for analysis. The reaction of calcium extraction is shown in reaction (R5.3).
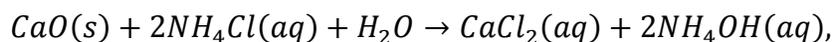
$$CaO(s) + 2NH_4Cl(aq) + H_2O \rightarrow CaCl_2(aq) + 2NH_4OH(aq),$$
$$\Delta H = -83 - 88kJ/mol \text{ (R5.3)}$$

The samples were diluted 10x and analysed with an ELIT Ion Selective Electrode (ISE). Table 5.1 shows the masses of Ca at the different times: numbers 1-2 represent the samples taken from the reactor at 60 minutes and 70 minutes, respectively, and numbers 3-4 represent the samples taken after the experiment from the reactor and from the second vessel, respectively.

TABLE 5.1 Ca^{2+} concentrations during and after extraction.

	1	2	3	4	
mol/L	0.057	0.058	0.078	0.065	(the dilution is taken into consideration)
g/L	2.285	2.305	3.106	2.601	(Ca: 40.08 g/mol)
g	45.691	46.092	62.124	52.024	(total mass of calcium dissolved in 20 liters)

When comparing these results with previous small laboratory experiment (chapter 3), one can notice that the concentration of Ca^{2+} is much higher than extraction in the larger scale experiment, see comparison in table 5.2. This is mainly because of the NH_4Cl solution in the settler, lowering the solid-to-liquid ratio of the total process to 60 g/L. A smaller Ca^{2+} concentration can also depend on the fact that the dissolution of calcium is much higher when using smaller particle size fraction [4]. In the small laboratory experiment, the particle size fraction consistently was 125-250 μm, while in the larger scale experiment the particle size fraction was 63-425 μm. Approximately 40% of the slag input was >250 μm.

TABLE 5.2 Ca^{2+} concentrations at different times during small scale and larger scale experiments

Extraction time (min)	20	40	60	70	100
Small scale, ~0.5 L (mol/L)	0.066	0.0805	0.0926		
Larger scale, ~20 L (mol/L)			0.057	0.0575	0.0775

Carbonation time (min)	8	15	30	45	55	60
Small scale, ~0.5 L (mol/L)		0.00623	0.00605	0.00671		0.00659
Larger scale, ~20 L (mol/L)	0.104	0.0763	0.0898		0.117	0.133

Samples taken from the carbonation step showed a higher Ca^{2+} concentration than in samples from extraction step, which was very unusual. It is possible that the ISE measurement contained an error. Due to this possible error, the conversion efficiency equation (E3.1) showed a negative result and apparently cannot be based on these ISE measurements.

5.4.3 Extraction and residual slag

After the termination of the experiment, the suspension in reactor, settling tube and second vessel, along with the residual slag were vacuum filtered using a Whatman filter paper (6 µm pores). The filtrate and the residual slag from the experiment were dried overnight at 100 $^{\circ}$C, and weighed thereafter.

Table 5.3 shows the obtained results; numbers 1,2,3 are solids from the suspension in the reactor, second vessel and settling tube, respectively, while numbers 4 and 5 are the residual slag solids from the settling tube and the reactor, respectively. No residual slag or settled particles were found in the second vessel due to the very efficient separation in the settling tube. The total mass after the experiment was approximately 1118.76 g, which is an 81.19 g difference from before the experiment (~1199.95 g) as a result of the extraction of mainly calcium.

Based on the obtained slag residue and slag input, the extraction efficiency was approximately 6.8%, which is 0.7% lower than the small laboratory batch experiment with similar slag and 14.2% lower than the semi-continuous laboratory experiment with steel converter slag (chapter 3).

TABLE 5.3 Mass of solids from suspension found in the reactor (1), second vessel (2), settling tube (3), and residual slag from the settling tube (4) and the reactor (5).

	1	*2*	*3*	*4*	*5*
Wet mass (g)	-	-	-	121.44	1012.00
Fluid (g)	-	-	-	2.65	21.90
Dry mass (g)	**2.16**	**1.81**	**5.9**	**118.79**	**990.10**
Mass-%	0.19	0.16	0.53	10.62	88.50

As expected, most of the residual slag found after the experiment was in the reactor, at approximately 992.26 g (including the filtrate) which is ~88.70 % of the total residual slag mass. Only approximately 1.81 g residual slag was found from a 10.4 L solution in the second vessel, which indicates a good particle separation from the suspension in the settler. It is, of course, desirable to obtain 0 g/L in the overflow but it is not realistic at this point. In future work, the overflow should be supplied with a filter to ensure and enhance the purity of the suspension.

Also a small change in the particle size distribution occurred as determined by sieving. Not suprisingly, when the slag particles dissolve in the NH_4Cl-solution, they become slightly smaller. When measuring the sizes after the experiment, it appears that some particles have also increased in size during the experiment. The initial size fraction before the experiment was 63-425 μm, but after the experiment approximately 3.11% of the particles were >425 μm, while approximately 0.51% were <63 μm.
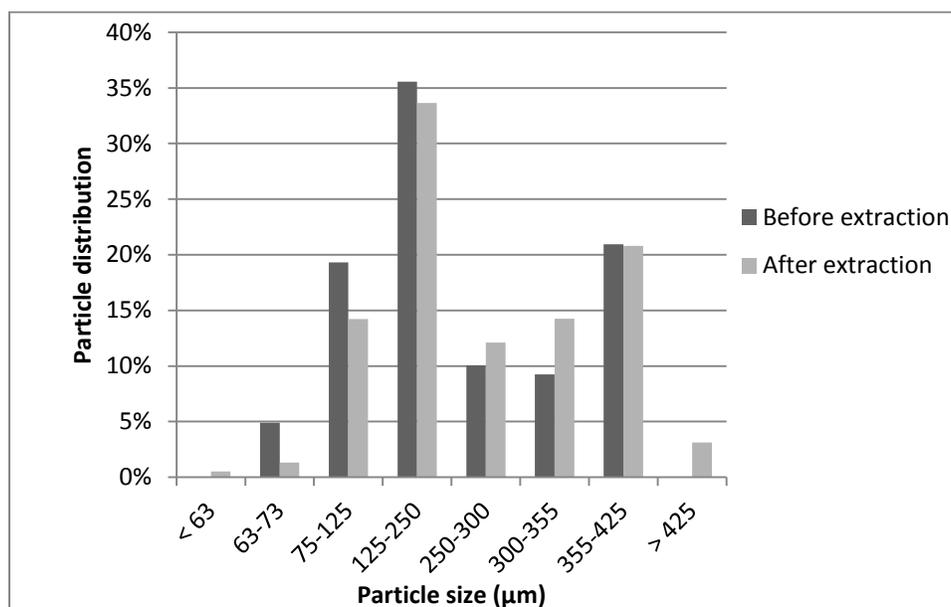
FIGURE 5.4 Particle size distribution (PSD) before and after extraction.

Approximately 5.02 g of the slag particles were smaller than 63 μm. This is also verified by a picture taken of the slag with a scanning electrode measurement (SEM). Figures 5.5 and 5.6 show that the particles from the overflow/second vessel are not larger than ~1-2 μm, while the residual slag particles from the reactor and settling tube have sizes <500 μm.
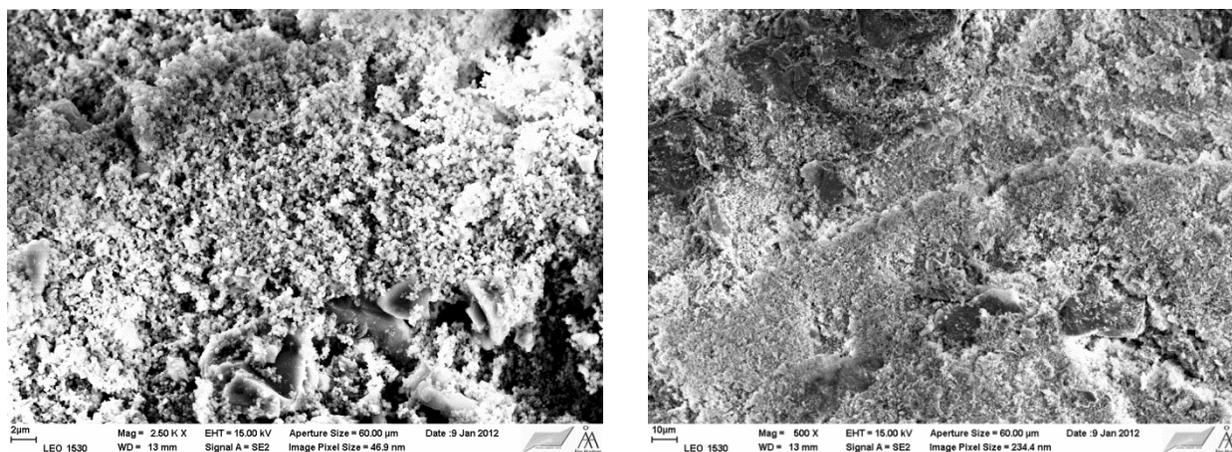
FIGURE 5.5 SEM pictures of particles from overflow.
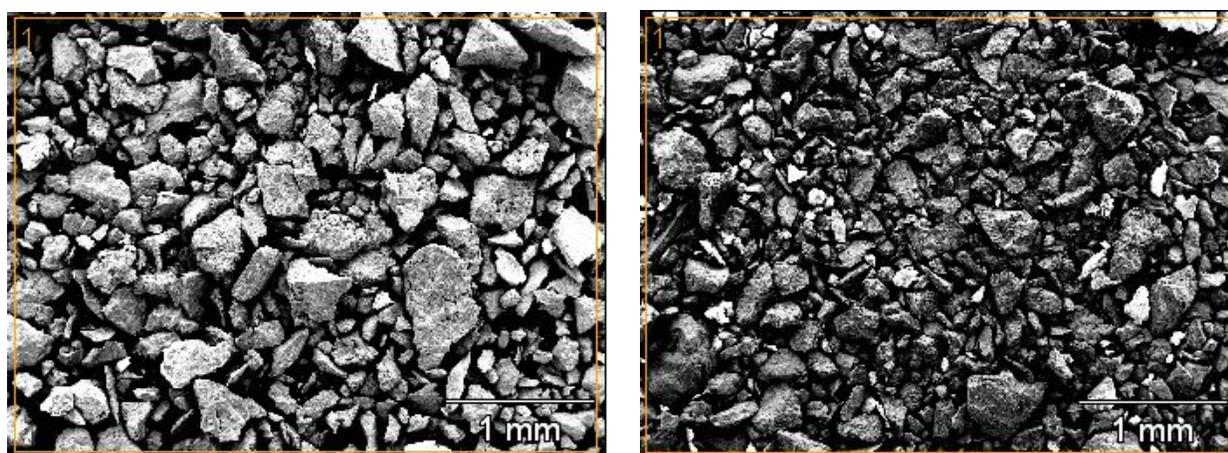
FIGURE 5.6 SEM pictures of the residual slag particles from reactor and settling tube.

5.4.4 Measured pH and temperature

The pH-value in the recirculated suspension from the overflow was measured with a Precisa pH900 every two minutes during the extraction, while pH in the reactor was measured continuously with an ISE. Figure 5.7 shows the changes of pH from both meters. The pH in reactor started to increase immediately after the slag was added, reaching its highest at 9.0 after 30 minutes. After 30 minutes, when the suspension was pumped into the settler, pH in the reactor dropped because of dilution with NH_4Cl and stabilized at approximately 8.8. The pH from the recycled overflow was not measured after 75 minutes when the stream was directed to the second vessel.
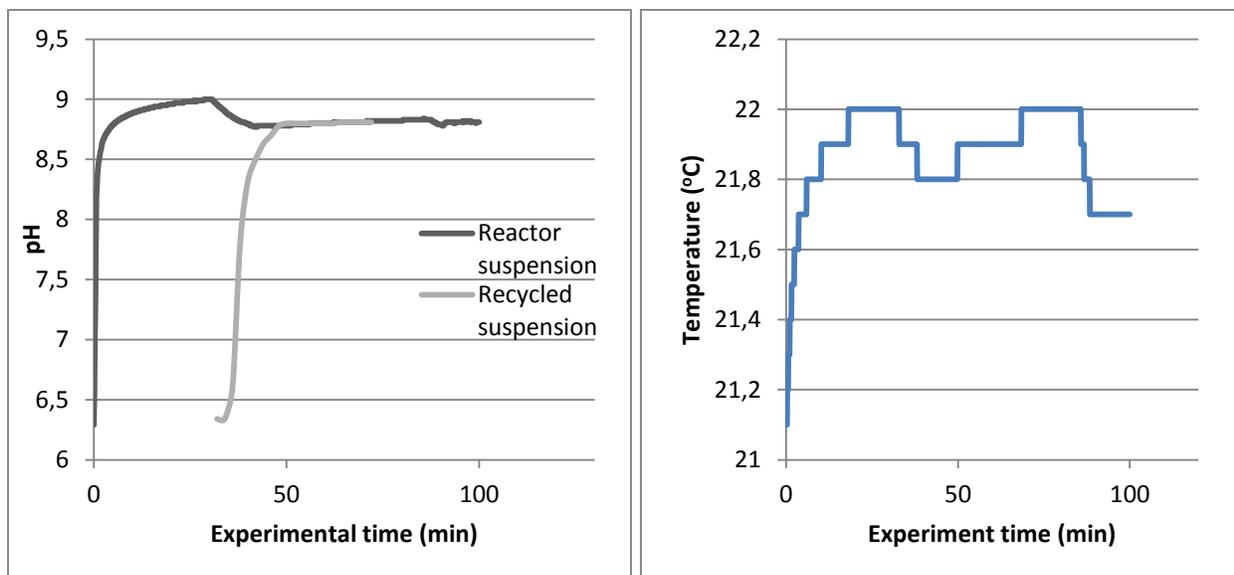
FIGURE 5.7 Measured pH (left) and temperature (right) changes during extraction. Pump started at t = 30 min.
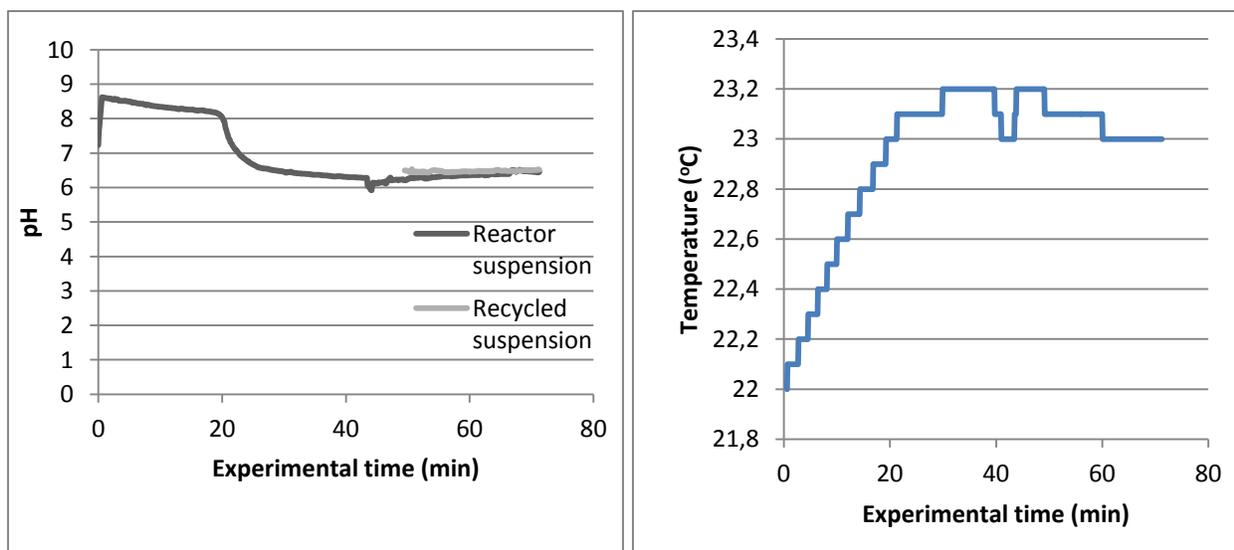
FIGURE 5.8 Measured pH (left) and temperature (right) changes during carbonation. Pump started at t = 40 min and recycling of suspension started at t = 50 min.

Figure 5.8 shows the measured pH and temperature changes during carbonation. The pH stabilizes at approximately 50 minutes at 6.5 when the recycling starts. The temperature increases from 22 $^{\circ}$C to 23.2 $^{\circ}$C during the first 30 minutes of carbonation. After the pump and recycling started the temperature ended at about 23 $^{\circ}$C.

5.4.5 Particle settling

During the experiments, the particle settling rate and the movement of the suspension were visually observed. The total possible travel distance for the particles and suspension was 110 cm and the top of liquid free surface was at a 77.8 cm height. During the extraction experiment, the separation of slag particles was started after 30 minutes, when the suspension entered the inclined settler. The suspension ascended in a rather rapid pace along the settling tube, reaching the top after 15 minutes of separation. The slag particle sediment ranges approximately 100 cm along the tube, which it reached after 35 minutes of separation, 65 minutes in the experiment.

Besides from some blockage in the underflow, another issue was observed. As a result of pump instability, occasionally air bubbles entered the settler with the feed flow, mixing the sediment and the suspension. The mixing resulted into a more turbid suspension, which made it more difficult to determine where slag particles had settled. In addition, there have been reports by others from similar experiments about instabilities inside the tube, lowering the efficiency of the separation [24].
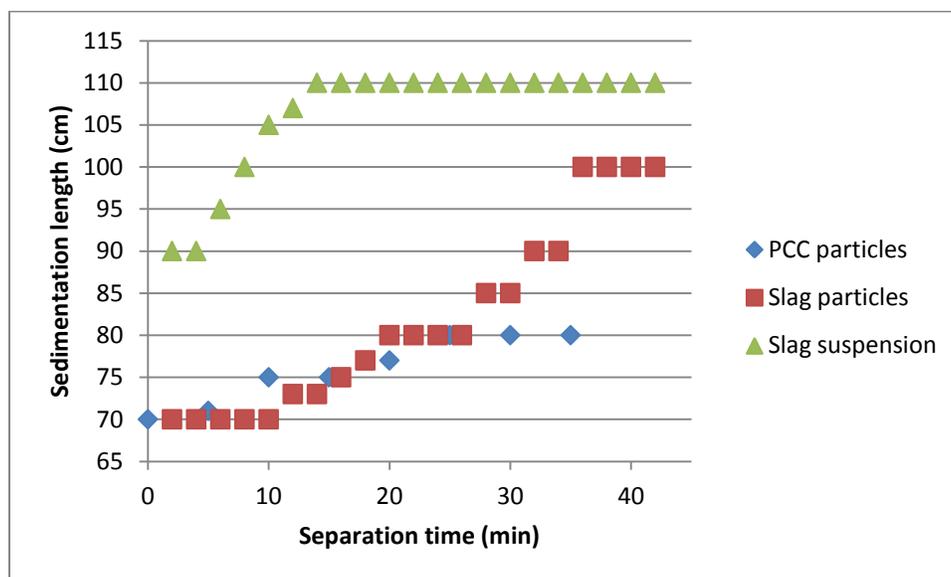
FIGURE 5.9 Particle and suspension movement during extraction and carbonation.

Separation of particles during the carbonation experiment was performed with some small differences from the extraction experiment. Unlike the extraction, the solvent was pumped into

the inclined settler first after the reaction with the CO_2-gas had started. Also, air bubbles in the feed flow, which caused some mixing of the sediment and suspension in the extraction experiment, could be prevented in the carbonation experiment.

Figure 5.8 shows the movement of the PCC particles along the settling tube compared with the slag particles. The movement of the suspension was not measured during the carbonation experiment. The sediment of PCC particles reached approximately 80 cm along the separator, 20 cm lower than with slag particles, even though the PCC particles are much smaller.

5.4.6 PCC particle concentrations

As mentioned, after approximately 40 minutes in the experiment the solvent was pumped into the inclined settler, and the separation started after 50 minutes in the experiment. For determining the particle volume concentration in the reactor, the PCC found in the samples from the underflow and overflow were also taken into account. The total measured mass of PCC found in the reactor and samples weighed approximately 48.11 g, the initial solvent volume in the reactor was approximately 10.55 dm^3, giving a particle volume concentration of c = 4.56 g/L.

During the experiment, samples were taken from both the underflow and the overflow at 65 minutes and 80 minutes. The underflow sample at 65 minute weighed approximately 9.45 g of 65 ml solvent, giving a concentration of $c_{u,65}$ = 145.4 g/L, or 14.5 % solid-to-liquid ratio, while the sample at 80 minutes weighed approximately 8.61 g of 45 ml solvent, giving a concentration of $c_{u,80}$ = 191.3 g/L, or 19.1 % solid-to-liquid ratio. The samples from the overflow weighed significant less than the underflow due to an effective separation. At 65 minutes, the sample from the overflow weighed approximately 0.01 g of a 62 ml solvent, giving $c_{o,65}$ = 0.16 g/L or 0.016 % solid-to-liquid ratio, while the sample from 80 minutes weighed approximately 0.12 g of a 1205 ml solvent, giving $c_{o,80}$ = 0.1 g/L or 0.01 % solid-to-liquid ratio.

However, these values do not satisfy the mass balance in equation (E4.1). The particle volume concentration in the reactor was approximately 4.56 g/L, which should also be the concentration of the feed. According to equation (E4.1), assuming c_{in} = 4.56 g/L and $c_{o,80}$ = 0.1 g/L, the particle volume concentration of the underflow equals 25.8 g/L, which is a great difference from the

experimental results. This could be explained by the fact that, similar to the extraction experiment, the underflow was occasionally blocked by the particles, which resulted into an irregular stream.

5.4.7 Separation efficiency

As the particle concentration was 84.45 g/L in the reactor and 0.17 g/L in the overflow during extraction, equation (E4.6) gives the separation efficiency for the extraction experiment to be $\eta = 99.8\%$. The particle concentration in the reactor was approximately 4.56 g/L and in the overflow about 0.1 g/L during carbonation, giving a separation efficiency of $\eta = 97.8\%$.

Extraction $\qquad \eta = (1 - \frac{0.17}{84.45}) \cdot 100\% = 99.8\,\%$

Carbonation $\qquad \eta = (1 - \frac{0.1}{4.56}) \cdot 100\% = 97.8\,\%$

The calculated carbonation separation efficiency is a few percent less than the results from the extraction experiment, although the overflow concentration was quite similar in both cases.

There are mainly two reasons why the separation efficiency is smaller in the carbonation experiment: (1) the PCC particles are much smaller than the slag particles and thereby settle at a slower rate, and (2) if the feed concentration would have been higher, the result would obviously have shown a different efficiency, assuming that the overflow concentration remained unchanged.

Due to the low terminal settling velocity of the smallest particles in the suspension it is doubtful that the separation efficiency of an inclined settler can be increased further. However, it is possible to optimize the overall process by e.g. shortening the settling tube. The chapter dimensional analysis (chapter 6) in this thesis presents a useful technique for how this can be done.

6 DIMENSIONAL ANALYSIS

Dimensional analysis is a useful method for reducing difficult mathematical problems to a more simple form to obtain a quantitative description. Bridgman explained dimensional analysis as "the principal use of dimensional analysis is to deduce from a study of the dimensions of the variables in any physical system certain limitations on the form of any possible relationship between those variables. The method is of great generality and mathematical simplicity [25]." Dimensional analysis can be applied in all experimentally based areas of engineering. If it is possible to identify the factors involved in a physical situation, dimensional analysis can form a relationship between them. This method was used already in works of great scientist of the eighteenth-nineteenth century, e.g. Fourier, Rayleigh and Reynolds.

6.1 Dimensions and units

Any physical event can be described by certain familiar properties, e.g. velocity, area, acceleration etc. All of these have dimensions. They are units for quantity, whose numerical values depend on the choice of base unit. A dimension is either fundamental or a derived dimension. Derived dimensions, like Pascal (Pa) and Newton (N), are products of powers of fundamental dimensions.

Each type of base quantity has by definition its own dimension. The complete list of the seven fundamental dimensions in The International System of Units (SI, Système International) are presented in table 6.1. All of the other dimensions for units are some combination of

.

$$[Q_i] = L^\alpha M^\beta T^\gamma I^\delta \Theta^\varepsilon N^\zeta J^\eta \tag{E6.1}$$

where the square bracket implies the dimension of the quantity of Q_i, the Greek letters are dimensionless numbers and the capital letters are dimensions of the SI system.

Quantity name and symbol	Dimension symbol	Unit name and symbol
length, *l*	L	metre, m
time, *t*	T	second, s
mass, *m*	M	kilogram, kg
temperature, *T*	O	kelvin, K
current, *I*	I	ampere, A
number of elementary particles, *n*	N	mole, mol
luminous intensity, I_v	J	candela, cd

As one can see in table 6.1, there are seven base quantities in the SI system: length, time, mass, temperature, current, number of elementary particles, and luminous intensity. The units for these are the metre (m), the second (s), the kilogram (kg), kelvin (K), ampere (A), mole (mol) and candela (cd), respectively. For matter either mass (kg) or amount in moles (mol) is used.

The purpose of dimensional analysis is to simplify a technical problem when, as in many cases, there are no mathematical descriptions or model equations available. Experimental work can be very expensive and solving complex equations can give too complicated solutions, or give no solution at all. Testing at a smaller scale and producing relations between the relevant parameters may give the information needed for successful scale-up.

6.2 Dimensional analysis procedure

The basic idea behind dimensional analysis is to compose a simple model that includes the important variables and parameters. The first step in performing a dimensional analysis is to determine all the relevant parameters that affect the system. A change in a relevant parameter will change the system. This is also the most important step; if an incomplete set of variables is chosen, the result will most likely be incorrect. A problem at hand can be expressed as

$$Q_o = f_1(Q_1, Q_2, .., Q_n) \qquad \text{(E6.2)}$$

where $Q_1,..,Q_n$ is a complete set of independent parameters and Q_o is the dependent parameter. The set is complete if no other quantity can affect Q_o and it is independent if the value of each quantity does not affect the value of another. Over-specification of independent variables does not disrupt the analysis, but it makes it less effective.

The next step is listing the dimensions of the independent variables $Q_1,..Q_n$ and the dependent variable Q_o. All physical quantities have dimensions which can be expressed as products of powers of the set of base dimensions.

Because all complete physical equations must be dimensionally homogenous and correct, a modification of an equation in a dimensionless form will reduce n independent quantities in by k fundamental units, and describing it by a relationship as n − k = m dimensionless groups; Π_1, $\Pi_2,..,\Pi_m$. This is called the Buckingham's Π-theorem [25]. This means that a relationship based on the (n) parameters Q is replaced by another relation based on a smaller number (n − k) of dimensionless groups.

$$\Pi_o = f_2(\Pi_1, \Pi_2, .., \Pi_m) \tag{E6.3}$$

The equation above does not tell us the forms of the dimensionless variables or the form of the functional relationship. It only tells us the number of dimensionless groups that affect the value of a particular dimensionless variable.

In practice, experiments are often conducted on a scale model instead of the real model. The scale model and the tests are geometrically and dynamically similar to the real model, but at a smaller size. If the variables from equation (E6.2) are determined from scale modeling, the result of the experiment can be directly translated to the real model. For this the dimensionless groups should have the same values for both scales.

6.3 Dimensional analysis for efficiency of inclined settler

A dimensional analysis for the inclined settler was performed according to the procedure given in the previous section. For determining the efficiency of the inclined settler, ten different parameters are considered to affect the result. The parameters are listed as

$$L_1 = f\left(L, b, Q_o, d_p, \rho_p, \rho, \mu, g, \sin\theta\right) \tag{E6.4}$$

where L_1 (m) is the length of the sediment, L (m) is the length of the separator, b (m) is the width, or diameter, of separator, Q_o (m^3/s) is the volume rate of the overflow, d_p (m) is the size of a single particle, ρ_p (kg/m^3) is the density of particle, ρ (kg/m^3) is the density of fluid, μ (kg/(m·s)) is the viscosity of the fluid, g (m/s^2) is the gravitational acceleration (~9.81 m/s^2), and θ is the angle of the inclination with respect to vertical.
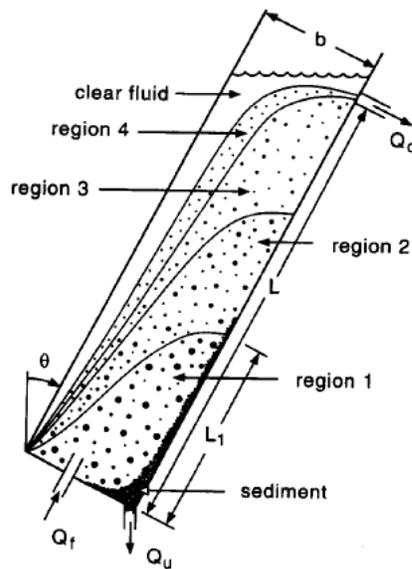
FIGURE 6.1 Schematic picture of inclined separator used in experiments [24].

A schematic picture of the inclined setter is shown in figure 6.1. In this work, the efficiency of the inclined settler is determined by the sediment length (L_1) inside the separator. As in most examples, the efficiency can also be determined by the difference of particle volume concentrations entering and leaving the separator, but due to poor concentration measurements of

overflows during experiments and good separation leading to zero particles in the overflow, concentration differences are not taken into account.

Parameters volume feed flow (Q_f) and underflow (Q_u) are not included in this dimensional analysis because the sedimentation rate is usually determined by the overflow volume flow.

No other parameter is considered to affect the system in equation (E6.4) and is, therefore, complete. The next step is to make the parameters dimensionless by using their base units. From these nine independent variables, three of the base units are present (kg, m, s), which gives six dimensionless groups ($n - k = m$; $9 - 3 = 6$) according to Buckingham's theorem. It should be noted that the inclination ($\sin \theta$) is already dimensionless and does not therefore need further transformation. To proceed with the dimensional analysis, the units are replaced with by a combination of basic units that characterizes the problem:

$$
\begin{aligned}
&m \to L \\
&kg \to \rho L^3 \qquad\qquad\qquad\qquad\qquad\qquad\qquad (E6.5)\\
&s \to \rho L^3 / \mu L
\end{aligned}
$$

The problem can now be expressed as

$$\frac{L_1}{L} = f\left(\frac{L}{L}, \frac{b}{L}, \frac{Q_o \cdot \rho L^3}{L^3 \cdot \mu L}, \frac{d_p}{L}, \frac{\rho L^3}{\rho L^3}, \frac{\rho_p L^3}{\rho L^3}, \frac{\mu L \cdot \rho L^3}{\rho L^3 \cdot \mu L}, \frac{g \cdot \rho^2 L^6}{L \cdot \mu^2 L^2}, \sin \theta \right) \qquad (E6.6)$$

$$\frac{L_1}{L} = f\left(1, \frac{b}{L}, \frac{Q_o \cdot \rho}{\mu L}, \frac{d_p}{L}, 1, \frac{\rho_p}{\rho}, 1, \frac{g \cdot \rho^2 L^3}{\mu^2}, \sin \theta \right) \qquad (E6.7)$$

$$\frac{L_1}{L} = f\left(\frac{b}{L}, \frac{\rho_p}{\rho}, \frac{g \cdot \rho^2 L^3}{\mu^2}\right) \cdot f\left(\frac{Q_o \cdot \rho}{\mu L}, \frac{d_p}{L}, \sin \theta \right) \qquad (E6.8)$$

$$\frac{L_1}{L} = C \cdot f\left(\frac{Q_o \cdot \rho}{\mu L}, \frac{d_p}{L}, \sin \theta \right) \qquad (E6.9)$$

In equation (E6.6) the independent variables, including the dependent variable, have been transformed into dimensionless groups by the suggested units in equation (E6.5). In equation (E6.8), by using the Buckingham's theorem, the number of independent variables has now been reduced to six. The first part of the function in the right-hand of (E6.8) contains dimensionless

groups that are constant at all times for a certain inclined settler, operating with certain particle density. The result shown in equation (E6.9) can also be expressed as

$$\frac{L_1}{L} = C \cdot \left(\frac{Q_o \cdot \rho}{\mu L}\right)^{\alpha} \cdot \left(\frac{d_p}{L}\right)^{\beta} \cdot (\sin \theta)^{\gamma} \qquad \text{(E6.10)}$$

where C is a constant.

For determining the values of the four constants (C, α, β, γ) in equation (E6.10) several experiments were conducted with an inclined settler. The settler had a total volume of 8.64 L (L = 1.1 m, b = 0.1 m) and was inclined in four different angles (0°, 10°, 30° and 45°) at five different volume feed flows (40 L/h, 45 L/h, 50 L/h, 55 L/h and 65 L/h). As the underflow was about 7 L/h in each experiment, the overflow was therefore Q_o: 33 L/h, 38 L/h, 43 L/h, 48 L/h and 58 L/h.

As particles, Ballotini glass beads and steel slag was utilized. Both 1 mol/L NH_4Cl-solution and water was used in different experiments, but in this case the properties ρ = 998 kg/m^3, μ = 0.001 kg/(m·s) are used. All experiments were performed in ambient conditions (~20 ^{o}C, 1 bar).

The experiments are more thoroughly discussed in chapters 4 and 5. The experimental results are also listed in the appendices.

For determining the optimal values for the constant and exponents in (E6.10), Windows Excel Solver was used for optimisation. The proposed values for (E6.10) are

$$C = 0.014$$
$$\alpha = 0.152$$
$$\beta = -0.337$$
$$\gamma = 0.37$$

$$\frac{L_1}{L} = 0.014 \cdot \left(\frac{Q_o \cdot \rho}{\mu L}\right)^{0.152} \cdot \left(\frac{d_p}{L}\right)^{-0.337} \cdot (\sin \theta)^{0.37} \qquad \text{(E6.11)}$$

The proposed values in model equation (E6.11) show a standard deviation equal to 0.18 from the experimental results. Due to a large difference of sediment lengths in the obtained experimental results, no direct correlation was found. It should also be emphasized that experiments with separator in 0° inclination were not included in the dimensional analysis because the results deviated too much from the other angles.

7   HYDROCYCLONE AS A SEPARATION ALTERNATIVE

From the current process particle separation using a hydrocyclone can also be considered as an alternative. The separation technique of a hydrocyclone is based on the effect of centrifugal forces resulting from pumping fluid tangentially into a cylindrical body. The hydrocyclone should not be compared to a centrifuge too much, since cyclones have no moving parts and the vortex motion is executed by the fluid.

As a result of a tangential entry, the fluid develops a swirling motion inside the cyclone. The particles dispersed the flow in a hydrocyclone are exposed to two forces; centrifugal force and drag force. These two forces are which are decisive for the performance of the cyclone; if the centrifugal force exceeds the drag, the particle moves outward, and if drag is greater, the particle is carried inwards [23].

Particle size which separates at 50% efficiency is referred to as the cut size, or cutoff size, and is often used to characterize the performance of a hydrocyclone. The underflow concentration of a hydrocyclone rarely exceeds 45-50% by volume, therefore a series or parallel connection of cyclones is a common way to enhance their separation efficiency [27].
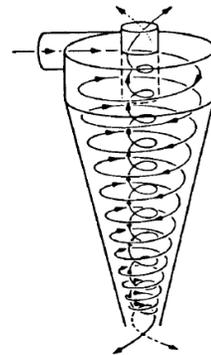
FIGURE 7.1 Flow principle inside a hydrocyclone [28].

Similar to sedimentation tanks, hydrocyclones are simple, easily installed and run and require very little maintenance. They are also compact, thus saving space and giving low residence time, which gives them an advantage in terms of the speed control. However, they are somewhat inflexible once installed and they give low turndown ratios due to the strong dependence of their separation performance on flow rate and feed concentration.

7.1 Hydrocyclone design and scale-up

The diameter of cyclones can vary from 10 mm to 2.5 m and the range of cut size for most particles is usually 2 to 250 µm. The flow rates for a hydrocyclone range from 0.1 to 7200 m^3/h and the pressure drop range from 0.34 to 6 bar [27].

Although several researchers have attempted to determine an empirical model for an optimal hydrocylone design, none have yet succeeded. However, a semi-empirical model has been developed by using three dimensionless equations (E7.1-E7.3). The dimensionless group implies that a given cut size and underflow concentration is known. Table 7.1 lists two well-known geometries and performance constants for narrow angle hydrocyclones.

TABLE 7.1 Geometries for hydrocyclones and correlation constants [27].

Cyclone type	D$_i$/D	D$_o$/D	D$_u$/D	1/D	L/D	Cone angle (deg)	Stk·Eu	K	n$_p$
Rietema	0.28	0.34	0.2	0.4	5	20	0.0611	316	0.134
Bradley	0.133	0.2	0.07	0.33	6.85	9	0.1111	446.5	0.323

The correlations derived from earlier studies [27]:

$$Stk_{50}Eu = 0.0474[\ln{(1/R_f)}]^{0.742}\exp{(8.96c)} \quad \text{(E7.1)}$$

$$Eu = 371.5Re^{0.116}\exp{(-2.12c)} \quad \text{(E7.2)}$$

$$R_f = 1218(D_u/D)^{4.75}Eu^{-0.30} \quad \text{(E7.3)}$$

where Reynolds number, Re, Euler's number, Eu, and Stokes number, Stk_{50}, (for cut size x$_{50}$) are defined as:

$$Re = \frac{vD\rho}{\mu} \quad \text{(E7.4)}$$

$$Eu = \Delta p / (\rho v^2 / 2) \tag{E7.5}$$

$$Stk_{50} = \frac{x_{50}^2 (\rho_s - \rho) v}{18 \mu D} \tag{E7.6}$$

The superficial velocity in the cyclone is used as the characteristic velocity:

$$v = \frac{4Q}{\pi D^2} \tag{E7.7}$$

The symbols are explained in table 7.2. The equations (E7.1-E7.3) represent a helpful model for hydrocyclone design and/or scale-up. The model allows the designer to select the best compromise between hydrocyclone performance and the design and operating conditions likely to achieve it.

TABLE 7.2 Nomenclature for symbols for chapter 7.

Symbol	Explanation
c	Particle concentration (kg/kg)
D	Diameter of cylinder (m)
D_i	Diameter of inlet (m)
D_o	Diameter of overflow (m)
D_u	Diameter of underflow (m)
L	Length of cyclone body (m)
Q	Feed flow rate (m^3/s)
Rf	Underflow-to-throughput ratio (flow ratio)
Stk_{50}	Stoke's number
Stk_{50}'	Reduced Stoke's number
v	Characterisitic velocity (m/s)
μ	Viscosity of fluid (Pa·s)
ρ	Density of fluid (kg/m^3)
ρ_s	Density of solid (kg/m^3)

The two hydrocyclone geometries given in table 7.1 have been tested for solid concentration up to 10 kg/kg [27]. For larger solid concentration the values in table 7.1 for the constants n_p and K

might not be valid. If one wishes to use hydrocyclones in parallel, the feed flow is divided by the number of cyclones in parallel.

K and n_p are empirical constants for geometrical similar cyclones that can easily be obtained from a number of experiments. Dimensional analysis gives two relationships between the three dimensionless groups as:

$$Stk_{50} \cdot Eu = constant$$

and

$$Eu = K \cdot (Re)^{n_p}$$

The group $Stk_{50} \cdot Eu$ is usually between 0.06 and 0.33, while the exponent n_p varies from 0 and 0.4.

7.2 Separation by hydrocyclone

A hydrocyclone can be a valid option for separating steel converter slag and PCC particles from aqueous solutions. Unfortunately, the operating volume flows in the experiments in this thesis were too small to be tested on a reasonably sized hydrocyclone. It is, however, possible to calculate the dimensions and the pressure drop for one or several cyclones for a given particle cut size.

Using the dimensionless group (E7.1-E7.3) and the cyclone geometries defined by Rietema, the necessary sizes and pressure drops for various volume flows can be determined for separating steel converter slag and PCC particles from a solution. The volume flows chosen are $1000 - 100\,000$ L/h and represent an actual industry scale process. As mentioned earlier, the range of volume flow for a hydrocyclone can be up to $7\,200$ m^3/h. The particle cut sizes are 10 μm and 20 μm, but should possibly be smaller for PCC particle separation. The flow ratio, R_f, is not known, but for the purpose of these calculations it was set at 0.1.

The remaining necessary parameters, density, viscosity, and particle concentration, ought to be similar to previous experiments in the Slag2PCC-project. The values for the fluid represent a 1 M ammonium chloride solution; density is $\rho = 1015$ kg/m^3 and the viscosity is $\mu = 0.00098$ Pa·s. The density for a single particle is $\rho_s = 2600$ kg/m^3, while the particle mass concentration was set at 10 %-wt.

7.3 Modeling results

7.3.1 Cyclone diameter

Calculation results for hydrocyclone diameters with cut sizes 10 µm and 20 µm are plotted in figure 7.2. The results were obtained by combining equation E7.1 with E7.2 for volume flows 1 000 – 100 000 L/h. One can also see the effect on the diameter when having a parallel connection.
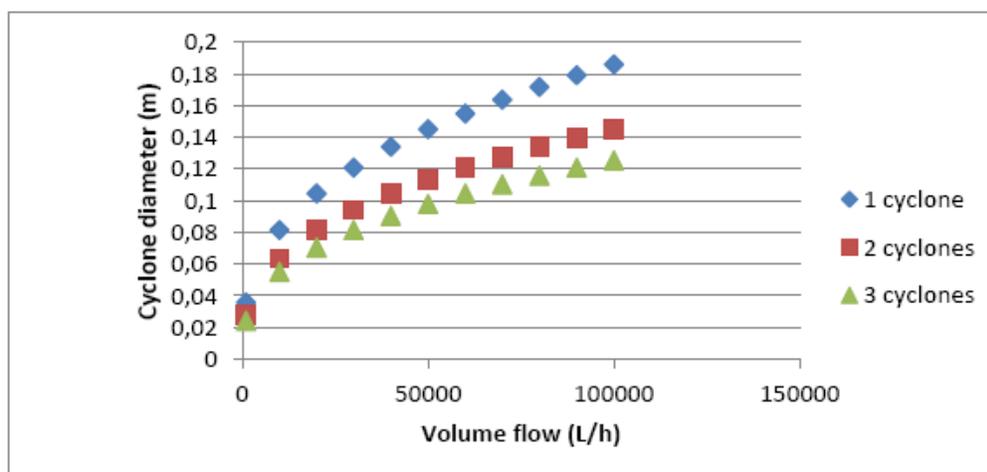
FIGURE 7.2 Hydrocyclone diameters for particle cut size 10 µm (upper figure) and 20 µm (lower figure).
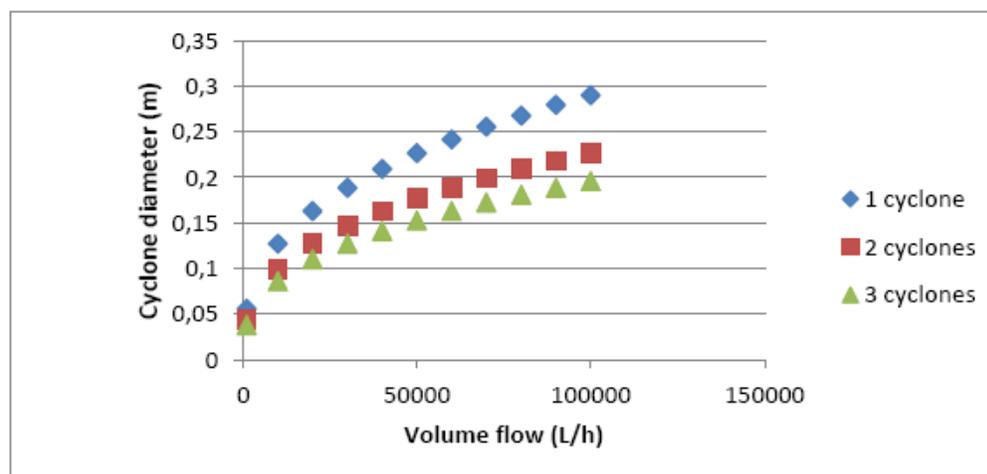
Naturally, the cyclone size increases with an increasing volume flow. The results reveal that even at 100 000 L/h volume flows for particle cut size 20 μm, the cyclone diameter is less than 30 cm. Cyclone diameter being 30 cm the total length of cyclone, including body and cone, would be about 1.8 m, using Rietema's geometries. Still, 1.8 m length and 0.3 m diameter is considerable smaller than a sedimentation tank at similar volume flows. As for comparison, using these dimensions (1.8 m length and 0.3 m diameter) in equation (E4.5) for a 45° inclined sedimentation tank the particle cut size would be about 216 μm.

7.3.2 Static pressure drop inside cyclone

The static pressure drop is calculated from equation (E7.5). For the pressure drop calculations, similar volume flow was used, 1 000 – 100 000 L/h, and the cyclone diameters are identical to the results obtained in the previous section. The results are plotted in figure 7.3.
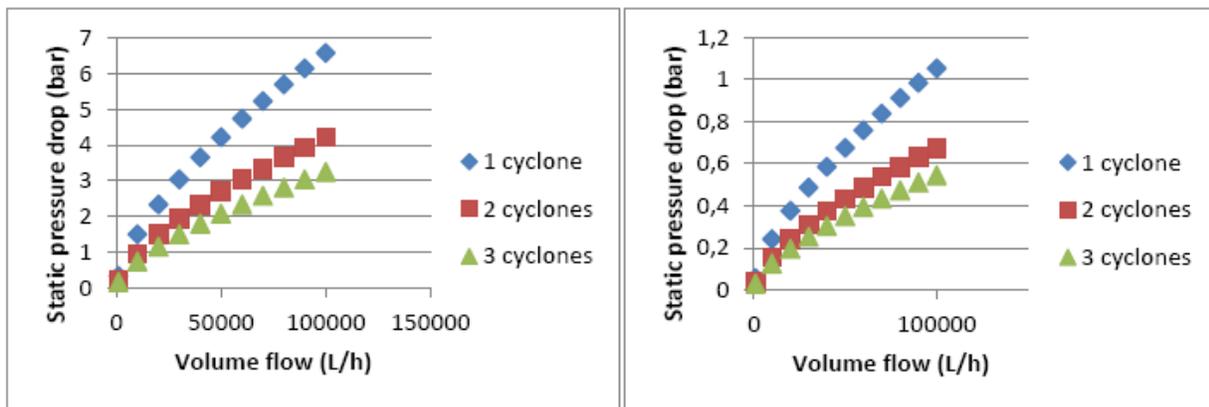
FIGURE 7.3 Static pressure drop inside cyclone for particle cut size 10 μm (left) and 20 μm (right).

As presented in figure 7.3, static pressure drops inside cyclones for particle cut size 10 μm are much higher than in cyclones for particle cut size 20 μm. Pressure drops being as high as they are predicted here, they will most likely be equipped with pumps to achieve a steady flow and particle separation.

Obviously, there are several other parameters that affect the performance of a hydrocyclone, which need to be taken into consideration. However, the purpose of this chapter is to give an

insight into using hydrocyclones instead of (inclined) sedimentation, a more thoroughly analysis and description of the performance should be done in a separate, more extensive study.

8   SUMMARY AND CONCLUSIONS

There is no doubt that the global carbon dioxide emissions are increasing at a high rate and powerful methods need to be presented for slowing this down. None of the current CCS methods are both economically and environmentally feasible, and the risks are considered to be high. However, progress was demonstrated in the field of mineral carbonation. Mineral carbonation includes calcium reacting with carbon dioxide producing solid calcium carbonates. Mineral carbonation is also a safer alternative (being leakage-free)than the other CCS-methods.

The Tekes (Teknologian ja innovaatioiden kehittämiskeskus) funded project Slag2PCC (followed by Climbus Slag2PCC Plus and currently Cleen CCSP) started in 2005 for investigating the possibility of carbonating calcium-rich steelmaking slags while simultaneously cover three different areas; reusing a low-value industrial waste, decreasing carbon dioxide emissions and producing precipitated calcium carbonate (PCC) for commercial use. These projects have been a collaboration between Åbo Akademi University and Aalto University with several industrial partners including the steelmaking company Ruukki, mineral and processing company Outotec and mining company Nordkalk.

Although extraction of calcium from steelmaking slag has given good results, it is still unsure if Ruukki will change their steelmaking process which then also affects the slag composition; the "new" slag was reported to contain more vanadium and less free calcium oxide. Performed experiments showed that extraction efficiency of calcium from conventional slag was ~20%, compared to ~7% from the new slag. Comparing these numbers for a continuous industry scale PCC process, a 25 t/h steelmaking slag processing would give approximately 5 t/h PCC from the conventional slag, while the new slag would result in 1.75 t/h PCC.

Before experiments for a larger-scale carbonation process were performed, continuous separation using an inclined settler was investigated. Spherical Ballotini glass beads and steel converter slag was utilized for these experiments. The experiments were performed at different volume flows and in different settler inclinations in ambient conditions. All experiments with glass beads showed good results; all particles settled in the settler, giving a separation efficiency of 100%.

Also, by inclining the settler, a circular perspex circular tube, the settling rate of glass beads showed a remarkable difference. At the highest volume inflow tested (~65 L/h) in 0° inclination the particle sediment ranged 70 cm, while in 10°, 30° and 45° inclinations the particle sediment ranged approximately 47.5 cm, 42 cm, 40 cm, respectively. The glass beads had a similar size fraction in each experiment (125-212 μm). At the lowest volume inflow tested (~40 L/h) in 0° and 10° inclinations the particle settled in the same range (~22 cm). The glass beads "traveled" a longer distance in the settling tube than predicted by theory, but it may depend on the pulsating inflow from the peristaltic pump.

In experiments with steel converter slag, the particles were sieved to 63-125 μm fractions using the settler at 30° and 45° inclinations, while the settler in a 10° inclination the particle size fraction was <125 μm. The volume inflow was adjusted at ~40 L/h in these three experiments with slag and the underflow was set at ~7 L/h. During these experiments some blockage occurred in the underflow which caused an irregular stream.

At 30° and 45° inclinations the steel converter slag formed an 82 cm and 80 cm, respectively, long particle sediment along the settling tube, while at 10° inclination particle sediment was formed along the entire settling tube (110 cm). Although the particle sediment reached the overflow orifice at 10° inclination, no notable difference in the particle concentration was measured as the overflow particle concentration was similar in all three experiments with steel converter slag (~0.13 g/L). The particle concentration of inflow was ~90 g/L during the experiments, giving a separation efficiency of 99.86%.

In the larger scale extraction and carbonation experiment the same newer slag as mentioned earlier was utilized, as was the perspex settling tube for continuous separation of solids. In these experiments the slag particles were sieved to a 63-425 μm size fraction, the volume inflow was set at 40 L/h and the settler was inclined at 45°. During the extraction the slag was separated from the fluid very effectively, the particles formed a 100 cm long sediment along the tube and the particle concentration of the overflow was only 0.17 g/L. As the particle concentration of the inflow was ~84.45 g/L, the separation efficiency resulted into 99.8%. Further analyses of the

overflow suspension showed that the particles were of sizes ≤ 2 µm. Particles this small settle at a very slow rate or may not settle at all.

Regarding carbonation, the separation of PCC particles was better than expected. The separation efficiency was approximately 97.8%, the overflow particle concentration being only ~0.1 g/L. The PCC particles traveled approximately 80 cm along the settling tube and SEM analyses showed that the particles were ≤ 30 µm and contained some chloride from the NH_4Cl-solution.

For future scale-up work a dimensional analysis for efficiency of inclined settler was performed based on experimental results. In this case, the efficiency was determined by the sediment length and not the difference of particle concentration entering and leaving the settler due to the very good separation found in experiments (c_o = ~0 g/L). However, it should be emphasized that the proposed values in the dimensional analysis show a standard deviation equal to 0.18 from the obtained results and experiments with settler at 0° inclination were not included.

The alternative of using a hydrocyclone for separating solids from a larger stream should be taken into consideration. Hydrocyclones are similar to sedimentation tanks as they have no moving parts and require very little maintenance. They are also smaller in size than sedimentation tanks, which can be considered as an advantage for larger streams. For a hydrocyclone with 1.8 m length and 0.3 m diameter in a 100 000 L/h volume flow the particle cut size would be about 20 µm, while the particle cut size for a sedimentation tank with similar dimensions would be about 216 µm.

The performed experiments reported in this thesis have showed that it is possible to separate steel slag and PCC particles from an aqueous solvent with gravity sedimentation. The separation efficiency clearly increased when the settler was inclined, as the particles "traveled" a shorter distance. However, the inclined settler can be further enhanced. Particles ≤ 2 µm settle in a very slow rate or may not settle at all, which suggest that the settler need to be applied with a filter for a better precipitation of calcium carbonate and to ensure the purity of the PCC particles. In addition, in each experiment with the inclined settler a blockage of particles occurred in the

underflow. For a better separation and easier regulation of the stream in the future, the underflow should be supplied with a pump.

The PCC particles also contained a small amount of chloride from the solvent, which indicates that the particles need to be washed. Water usage for washing of particles in a continuous process needs further research.

Although there are some areas in the Slag2PCC-process that need improvements, research has so far shown good results. In the near future, this two-stage carbonation process will be scaled up to 100 kg/h at Aalto University in Espoo with support from Åbo Akademi University, which indicates that the project is progressing to a full industrial scale process.

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Appendices: **Appendix 1:** Experimental results

Material	Density (kg/m^3)	Size fraction (µm)	Volume, in (L/h)	Volume, u (L/h)	Volume, o (L/h)	Angle ^{o}	c,in (g/L)	c,o (g/L)	Sediment L$_1$ (cm)		L$_1$/L
Ballotini	2480	125-212	40	7	33	0	90		22		0,2
Ballotini	2480	125-212	45	7	38	0	90	0	40		0,364
Ballotini	2480	125-212	50	7	43	0	90	0	54		0,491
Ballotini	2480	125-212	55	7	48	0	90	0	62		0,564
Ballotini	2480	125-212	65	7	58	0	90	0	70		0,636
Ballotini	2480	125-212	55	7	48	30	90	0	41,5		0,377
Ballotini	2480	125-212	65	7	58	30	90	0	42		0,382
Slag	2604	63-125	40	7	33	45	90	0,127	80		0,727
Ballotini	2480	125-212	45	7	38	45	90	0	37		0,336
Ballotini	2480	125-212	50	7	43	45	90	0	38		0,345
Ballotini	2480	125-212	55	7	48	45	90	0	39,5		0,359
Slag	2604	<125	40	7	33	10	90	0,129	110		1,000
Ballotini	2480	125-212	40	7	33	10	90		22		0,200
Ballotini	2480	125-212	45	7	38	10	90	0	38,5		0,350
Ballotini	2480	125-212	50	7	43	10	90	0	44,5		0,405
Ballotini	2480	125-212	55	7	48	10	90	0	45		0,409
Ballotini	2480	125-212	65	7	58	10	90	0	47,5		0,432
Slag	2604	63-125	40	7	33	30	90	0,129	82		0,745
Ballotini	2480	125-212	45	7	38	30	90	0	36		0,327
Ballotini	2480	125-212	50	7	43	30	90	0	41		0,373
Ballotini	2480	125-212	65	7	58	45	90	0	40		0,364
Extraction	2720	63-425	40	7	33	45	84,45	0,17	100		0,909
Carbonation	2600	10	40	7	33	45	4,56	0,1	80		0,727

Appendix 2: Sweco hydrocyclones

SWECO Hydrocyclone

Inch	Median Micron Cut	Litre/Minute
4	15-20	225 to 250
5	20-25	300 to 400
10	40-45	2250 to 2500

The following rules are the most important factors to observe when operating a hydrocyclone :

1) The hydrocyclone operates best at specific fluid pressure (which is directly related to the amount of fluid and the fluid weight)
2) To operate a hydrocyclone efficiently it is important to control the fluid pressure at the inlet of the hydrocyclone on the inlet manifold
3) The amount of fluid processed should be a result of the correctly adjusted fluid pressure and not the result of desired fluid rate
4) There are two types of solids in respect tot hydrocyclone performance
   a) Separable solids
      solids the hydrocyclone can separate
   b) Non separable solids
      solids too small to separate
      or too light to separate
   Separation of solids is the result of centrifugal force on the weight of a particle (the combination of the factor specific gravity and the factor size rotating in a fluid)
5) The weight difference of the particle to be separated is directly related to the weight of the fluid (if both are equal, separation of particle will not result)
6) The solid trust be higher in weight and as larger the difference as higher is the separation efficiency (reversed separation is mechanically not possible)
7) The fluid/separable solid ratio is :

Hydrocyclone	Ideal	Maximum
4 inch	2 %	5 %
5 inch	2 %	5 %
10 inch	4 %	8 to 10 %
(Sweco Only)	By volume	By volume

There is no minimum.
<u>Note :</u> a well-designed hydrocyclone will discharge only very little or no water when there are no separable solids in the fluid

8) If the separable solids content exceeds the maximum, dilution is necessary
9) Overflow manifold must be venture except with an open or large and angled 45° effluent discharge
10) The discharge is generally 50 % solids/50 % fluid by volume (very high S.G. solid will change the solid/fluid ratio marginally)

Hydrocyclone Operation

1) Hydrocyclones operate well between 1 % min. and 4 % max. separable solids (best at 2 % separ. solids)
2) Separable solids are those which the hydrocyclone is designed to remove
3) Solids are removed by centrifugal force. The slurry spines in a bottom ward spiral along the outer wall and the heavier solids are pushed outward and build the outward layer falling through the bottom opening or solids discharge. The fluid trumps upward at the bottom and is discharge at the top centre.
4) Separation of solid in a hydrocyclone is a result of the specific gravity and particle size. As larger the specific gravity difference between the fluid and the solids as finer particles can be separated. If fluid and solids have the same specific gravity, than solids will not separate from the fluid.
5) Viscosity of the fluid has an effect on the separation process, viscosity will retard separation since it effects the speed with witch the solids can travel through the fluid to the outside (layer) of the slurry.
6) Particle size relative to specific gravity of the solid will effect separation efficiency most hydrocyclones are rated at the specific gravity of same or limestone more or less or at a specific gravity of 2.5 sp.gr.
   Example : same at 2.2 sp. Gr.
   Barite at 4.5 sp.gr.
   The same hydrocyclone will separate a barite particle four times smaller than a sand particle at the same rate
7) Separation median cut

Hydrocyclone size in inch	4 inch	5 inch	10 inch
Median cut in micron	15-20	20-25	40-45
G P M	54-56	12-18	540-560
Pressure PSI	35-45	30-40	16-25

8) Therefore → (1) and (6-9)
→ (1)   4 % solids by volume is the maximum efficient removal concentration of the slurry
→ (6-9)   particle size is a key factor of solids removal
            therefore is the solids concentration
Exceeds 4 % the hydrocyclone will remove the largess particles first and the finer but separable solids will remain in the fluid. The hydrocyclone will become inefficient.
   a) This situation may result in plugging
   b) The hydrocyclone will wear out much faster
9) In the case of slurries exceeding 4 % of separable solids by volume dilution is the correct procedure.
   Example : if the concentration is 6 % separable solids by volume dilute the slurry at least 50-50 or best dilute to 2 % solids maximum. This can be done as shown in the sketch. Dilute to the correct % solids in the feed tank and than use the necessary amount of hydrocyclones to maintain dilution.

<u>Example</u> : Processing of 100 GPM slurry with 10 % solids by volume. Particle size 25 to 45 µ of sand like material, sp. Gr. 2.5 in water viscosity 10 c.p.
→ dilute to 2 % solids by volume = 500 GPM
→ use 7 x 5 inch hydrocyclones if the slurry is constant 10 % or less concentration use 8 x 5 inch hydrocyclone if the slurry is 10 % or marginally higher concentration.

10) Since a good hydrocyclone is the most cost effective separation process (except in the dessert) it would be the incorrect approach to save hydrocyclone investment, or pump cost eventually an improper installation will be by far more costly. The life of a well-designed polyurethane hydrocyclone is bottom come 1 year top come or fluid splitter 10 year on the average.

11) In some cases hydrocyclone increasing operation pressure can increase efficiency. This must be done with great caution though. Increased pressure will potentially increase hydrocyclone wear. In general keep the operation pressure as recommended especially with sharp and or abrasive particles.

12) With increase processing temperature the hydrocyclone wear

13) Note only for Sweco Personal

The Sweco 10" hydrocyclone is one of the finest in the world it has been copied by our best competitors.

The 5" hydrocyclone is an excellent hydrocyclone with a median cut better than 20 micron in most cases at 2.5 sp. Gr. And 10 cp.

The 4" hydrocyclone is very good but needs a modification at the next redesign.

14) Bottom come adjustment or discharge insert press is used to balance the separation efficiency. The procedures, however, is only effective if
   a) The amount of fluid pumped is constant (GPM constant)
   b) The solids content is constant and variations in size and specific gravity of the solids is constant
   c) If the viscosity of the slurry is constant and no large temperature Variation exists.

15) The largess particle in the slurry should be less in it's largess dimension than ¼ of the orifice opening (including the bottom insert

   Hydrocyclone   orifice large particle   pigment
   4 inch
   5 inch
   10 inch

PSi × 0,07 = BAR
$\frac{m^3/h}{60} \times \frac{1000}{3,78}$ = GPM.

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SWECO HYDROCYCLONE SIZING CHART
CAPACITY/PRESSURE/SEPARATION = 71°C

Sizing based on water at maximum temperature 160° F. containing sand and grit (2.65 sp. g) not to exceed 25% by weight. Particle distribution must be fairly uniform top to bottom without a significantly large amount of paint-like slime. Pressure reading is at inlet of Hydrocyclone. Overflow and underflow are unobstructed and discharge to atmosphere. Overflow will contain a maximum of 5% of the plus "mesh of separation size" and approximately 50% of the minus "mesh of separation size." Underflow will have a concentration of 50-60% solids by weight. It will contain 95+% of the plus "mesh of separation size" and approximately 50% of the minus "mesh of separation size." No attempt has been made to show all of the combinations of sizes possible for a given flow rate such as shown for 45 and 50 gpm as an example.

GPM	Cyclone Size	Inlet Press. PSI	Mesh of Sep.	GPM	Cyclone Size	Inlet Press. PSI	Mesh of Sep.
25	4"	10	325	460	10"	23	325
30	4	15	325	480	10	25	400
35	4	20	325	500	10	27	400
40	4	25	400	520	10	30	400
45	4	30	400	540	10	31	400
45	5	11	325	560	10	33	400
50	4	40	400	580	10	35	400
50	5	14	325	600	10	37	400
55	5	16	325	650	19	10	200
60	5	19	325	700	19	12	200
70	5	25	400	750	19	13	200
80	5	35	400	800	19	15	200
90	5	40	400	850	19	17	200
100	2-5	14	325	900	19	19	200
110	2-5	18	325	1000	19	22	200
120	2-5	20	325	1100	19	26	250
140	2-5	25	400	1200	19	30	250
160	2-5	35	400	1300	19	35	250
180	2-5	40	400	1400	19	40	250
200	3-5	25	400	1400	28	8	150
220	3-5	30	325	1500	28	9	150
240	3-5	35	325	1600	28	10	150
260	3-5	40	400	1700	28	11	150
280	10	10	325	1800	28	12	150
300	10	11	325	1900	28	13	150
320	10	12	325	2000	28	15	150
340	10	14	325	2100	28	16	150
360	10	15	325	2200	28	17	150
380	10	17	325	2300	28	19	150
400	10	18	325	2400	28	20	150
420	10	19	325	2500	28	22	150
440	10	21	325	3000	28	30	200

POLYURETHANE HYDROCYCLONES
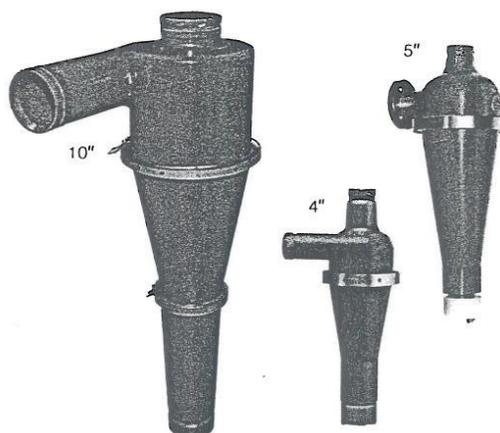
SWECO's polyurethane hydrocyclones offer drilling contractors an economical alternative to conventional mud cones. They are low-cost, easy to service and lightweight. And, they last over twice as long as the wear parts in conventional cast iron cones.

SWECO's entire cone sells for about the same as the replacement wear parts for a conventional cast iron cone. And replacement requires substantially less labor.

Our cones are molded from polyurethane because of its superior resistance to abrasion. But there are other advantages to polyurethane — high impact resistance, low weight and excellent resistance to chemicals. This makes polyurethane the ideal material for mud cone construction.

SWECO 4-inch desilter cones retrofit desilter manifolds with grooved connections. SWECO 5-inch desilter cones retrofit conventional desilter manifolds designed for 4-inch high capacity cones. SWECO 10-inch desander cones fit SWECO desanders.

SIZE	WEIGHT	CAPACITY
4"	9¼ lbs	50 gpm @ 30 psi
5"	14¼ lbs	70 gpm @ 30 psi
10"	74 lbs	500 gpm @ 30 psi

HOW IT WORKS

Overflow
Liquid containing some fine or light solids.

Feed
Pressurized slurry enters chamber in circular laminar flow.

Liquid with light or fine solids fraction moves inward in a spiralling vortex discharging upwards around an air core.

Pressure energy is converted to centrifugal forces driving solids outward and downward along conical wall in a spiralling flow.

Underflow
Removed solids with small amount of liquid.

ABRASIVE-RESISTANT LINERS

SWECO's specially formulated abrasive-resistant polyurethane* provides excellent internal wear resistance. For highly abrasive slurries, SWECO Hydrocyclones can be ordered with bonded inserts of fired alumina, or the newest silicon carbide in the lower-cone and apex areas. Alumina and silicon carbide liners are imbedded in a thick backing of urethane for cushioning and positive alignment.

APPLICATIONS

Include classification, degritting, and desliming operations in pulp and paper, sewage treatment, chemical, food, nonmetallic, and metallurgical processing — especially appropriate for closed-circuit grinding systems. A special sand separator is also available for removing small amounts of very fine sand from municipal and industrial water systems discharging a minimum of water with the sand.

SWECO CAPABILITIES

SWECO has been a leader in supplying equipment for liquid/solids separation for over 25 years. Application Engineers are available throughout the country to analyze your requirements and recommend the best solution. Computerized solids-removal profiles enable SWECO to convert your slurry characteristics quickly into anticipated separations and provide you with a material balance printout. A Hydrocyclone or a manifolded Hydrocyclone system alone may suffice, or you may require a more complete system combining other SWECO components. SWECO designs and manufactures individually tailored systems incorporating with the Hydrocyclone its Vibro-Energy Separator, Centrifugal Screen Concentrator, Sta-Sieve or Vibro-Energy Grinding Mill.

*SWECO's 16 years of experience in processing urethanes insures the latest, most cost-effective abrasion-resistant linings for its cyclones, grinding mills and finishing mills. Production procedures are continually being updated to incorporate the latest technical advances in urethane formulation and casting.

All specifications are subject to change without notice

Bottom: System as installed at C.L. Pharris Sand and Gravel Company, Corona, California

SWECO 4" & 5"
HYDROCYCLONE