Original Research Article

Small Pilot System for the Manufacture of In Situ Precipitated Calcium Carbonate in the Presence of Pulp Fibers

ABSTRACT

Paper fillers materials are less expensive than fiber, allowing reduced production costs and improvement of paper properties
A small pilot scale in situ laboratory calcium carbonate filler precipitation unit was designed with the objective to provide enough pulp fiber containing insist precipitated calcium carbonate for a small laboratory paper machine. The system developed system was tested with eucalyptus pulp fibers.
The final filler content achieved was 38.2%, 55.5% and 66.6% based on initial eucalyptus pulp fiber content. The precipitation time from an initial pH of 12.77, 12.76 and 11.98 to an final pH 7.29, 7.55, and 7.28 for the 3 kg, 6kg, and 9 kg of calcium hydroxide was 45 minutes for the 3 kg and 9 kg calcium hydroxide addition and 40 min. for the 6 kg calcium hydroxide addition.

Keywords: Calcium carbonate, eucalyptus, filler, in-situ precipitation, hybrid filler, paper

1. INTRODUCTION

Today, the paper industry around the world is experiencing significant challenges due to stringent environmental laws; globalization, high competitiveness and substantial pressures on profit margins [1]. The global paper product market stood at roughly 420 million metric tons in 2018 [2]. Paper products require worldwide over 8 million tons of filler material [3].
Over the last decades, paper industry research has attempted to find the upper bound of calcium carbonate based ($CaCO_3$) filler content in a sheet of paper. Especial for printing and writing grades filler material has become a prerequisite in a the global paper product market [4]
The major paper fillers used in papermaking are ground calcium carbonate (GCC) and precipitated calcium carbonate (PCC). PCC is the largest category of filler in North America, with nearly 70% of the market share. The second most common filler type, with a market share of 15%, is Kaolin, followed by ground calcium carbonate (GCC) with 13%. Titan dioxide's estimated market share is about 2%. The use of Silica / Silicates accounts for 0.3% and Talc and Aluminum Trihydrate together account for approximately 0.1% [5].
Paper fillers materials are less expensive than fiber, allowing reduced production costs, improved optical paper properties, dimensional stability, and better sheet formation and printability. The use of fillers, especially PCC, is mainly driven by production cost issues and

the printing industry's need for higher quality, brightness and improved printability as well as increased machine speed on the paper machine and coating application side [6]. Applying filler material causes a decrease of the paper's strength [7]. On the negative side, adding filler material to a paper product decreases paper strength indicates a lower quality paper [7].

However, economic advantages are higher due to a savings potential to safe up to $ 4.0 for each 1% increase in filler content [6].

The use of $CaCO_3$ filler can be traced back to the1920's to U.S. Patent 1,372,193 and 1,328,299 were the use of ground and precipitation $CaCO_3$ for industrial uses were granted as low cost paint filler and pharmaceutical agent [8,9]. In the paper precipitated calcium carbonate (PCC) was implemented in the 1970's in Europe and in the 1980's in North America as the industry shifted from acid to alkaline papermaking process [10].

PCC is at present time applied in slurry or powdered form to the papermaking suspension before the sheet forming process occurs. The PCC filler material is manufactured in separate processing plants without pulp fibers. The following research project has the objective to design and test a multi-use laboratory precipitation unit that allows to produce up to 12 kg of pulp fiber material with in situ precipitated filler material. This would allow to operate a small 12" pilot plant paper machine running at a maximum speed of 2 m/min producing up to 4 kg of paper per hour for a detailed paper property investigation.

2. MATERIALS

For this research project producing In-Situ Precipitate Calcium Carbonate (SIPCC) hardwood pulp in the form of Bleached Eucalyptus Kraft Pulp (BEKP) from CMPC Celulosa was used. Calcium hydroxide ($Ca(OH)_2$) powder was obtain from Lhoist North America. Industrial grade carbon dioxide (CO_2) gas was used with a 99% purity, supplied in a pressurized container containing 50 lbs. (22.68 kg) of gas.

2.1. PRECIPITATION SYSTEM SET UP

Based on preliminary experiments at the 10 liter scale a Large Scale Laboratory Precipitation (LSLP) system was designed as shown in Fig. 1. The LSLP system consist of a 1000 l (264.17 gal) open stainless steel tank (1) that can hold up to 920 l pulp fiber suspension (2). A 0.55kW (0.75 hp) industrial tank mounted mixer (3) with a 3 blade propeller having 250 mm (10 inch) in diameter is installed for mixing the pulp suspension. A 0.75 kW (1 hp) transfer pump (4) is used for recirculating the pulp suspension through the Static Mixer (SM) (6) with an approximate volumetric flow between 56 l to 112 l/min (15 and 30 gal/min). From Carbon dioxide (CO_2) storage tank (7), CO_2 is transferred into a CO_2-heater (12) to maintain a CO_2 temperature between 20°C and 30°C. CO_2-heater (12) contains a 9.5 mm (.38 in) inside diameter copper coil emerged in a heated H_2O bath maintained at 60°C. The temperature and pressure of the CO_2 is measured with temperature probe (13) and pressure probe (14). Operation pressure is adjusted above the pressure indicated on pressure gauge (15) using the regulator of the CO_2 storage tank. From the CO_2-heater the CO_2 gas is added into the static mixer assembly (6) using a 0.5" clear PVC hose. Six Sulzer SMF mixing elements with 25 mm in diameter are used in the static mixer assembly (6) to mix the pulp suspension and CO_2 without causing plugging. All piping for the LSLP system was manufactured using Schedule 40 Polyvinyl Chloride (PVC) piping (5) with 1.5 in (38.1 mm) inside diameter. Recirculation flow is adjusted with 1.5 in PVC ball valves (6). Container (8) with agitator is used to prepare the 20% The $Ca(OH)_2$ suspension. The suspension is metered into tank (1) with a 20% solids content using a 0.5 in PVC Schedule 40 PVC pipe and 0.5 in PVC ball valve (9). A temperature probe (10) and pH probe (11) allow monitoring the in-situ precipitation of calcium carbonate in tank (1). Transfer pipe (16) allows to transfer the pulp fiber suspension to the next process step.

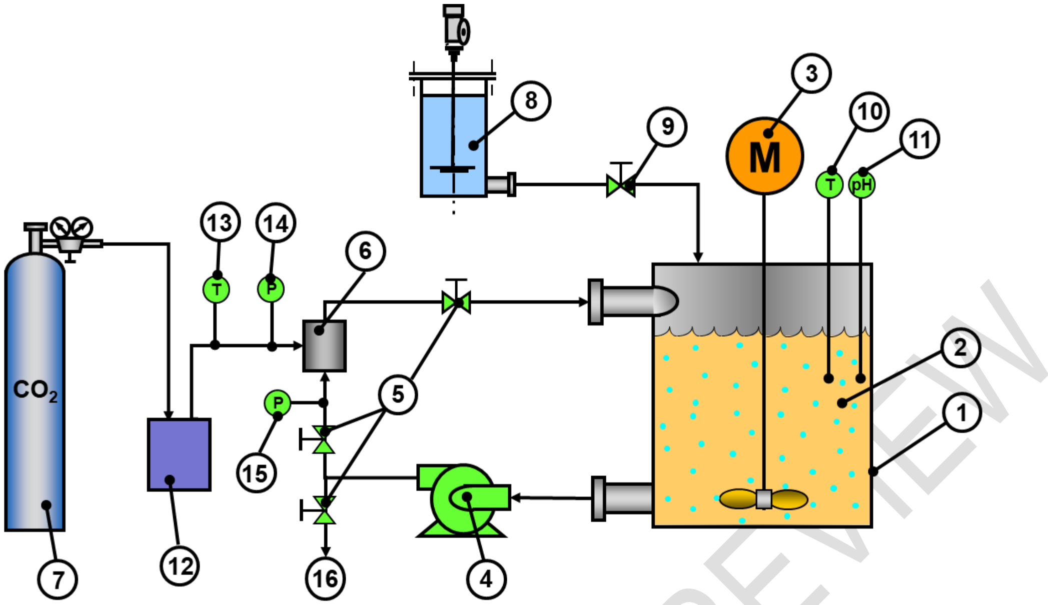

Fig. 1.: Large Scale Laboratory Precipitation (LSLP) system: 1) 1000 l tank, 2) Pulp fiber suspension, 3) 0.375 kW propeller mixer 4) 0.75 kW impeller pump, 5) Ball valve, 6) Static mixer, 7) CO_2 Tank with pressure transducer and flow adjustment, 8) lime tank, 9) Dosing valve, 10) Temperature Probe, 11) pH Probe, 12) Gas heater, 13) Gas temperature probe, 14) CO_2 pressure Gauge, 15) Pressure gauge, 16) Transfer pipe [11]

2.2. Operation of Large Scale Precipitation System

To produce ISPCC, first pulp fibers, in our case EC fibers, are pulped at a consistency of 5% using a laboratory pulper. The pulp fibers are then transferred into the LSLP system tank (1) and diluted to a consistency of 1.70% by adding H_2O to into the tank (1). Second, a $Ca(OH)_2$ suspension with 20% solids content is added from the lime tank (8) based on the final targeted ISPCC solids content level. Third, variable speed propeller mixer (3) is used to mix the resulting suspension of pulp fibers and $Ca(OH)_2$. Pulp suspension flow in tank (1) was adjusted in that way that good recirculation of the pulp suspension is ensured by an mixing vortex indentation in the center of 100 mm (4 in) on the tank surface. The mixing propeller (3) is kept in operation for the whole precipitation process to ensure good mixing of the pulp suspension and the precipitated ISPCC. After approximately 5 minutes of mixing recirculation pump (4) is started and the pulp suspension is recirculated with about 70 l/min (18.5 gal/min). Fourth, the pressure regulator of the Carbon dioxide (CO_2) storage tank (7) is opened and the CO_2 gas flow adjusted to 60.0 cfm (1.7 l/min) and a pressure of 68948.0 Pa (10.0 psi) above the pressure reading on pressure gauge (15) using pressure gauge (14). The CO_2 gas exiting the CO_2 storage tank (7) transfers into a CO_2-heater (12) to maintain a CO_2 temperature between 20°C and 30°C. The temperature of the CO_2 is measured with temperature probe (13). CO_2 gas exiting the CO_2-heater is then transferred into the static mixer assembly (6) using a 0.5" clear PVC hose. Six Sulzer-SMF mixing elements with 25 mm in diameter are used in the static mixer assembly (6) to mix the pulp suspension and CO_2 without causing plugging. The LSLP system is operated this way and temperature and pH values are monitored with probes (10) and (11) respectively. The LSLP system operation in stopped when pH of the recirculated pulp fiber suspension reaches a value of 7.0. The pulp fiber suspension with the ISPCC is then transferred into a receiving tank using transfer line (16) for further processing.

2.3. Testing Methods

Ash content was analyzed by TAPPI T211 om-02, "Ash in wood, pulp, paper and paperboard: Combustion at 525°C" [12]. Handsheets for physical testing were prepared in accordance with T 205 sp-06, "Forming handsheets for physical tests of pulp" [13]. Moisture contents were analyzed according to TAPPI T412 om-06, "Moisture in pulp, paper and paperboard" [14]. Measuring temperature and pH of the pulp suspension an Accumet AP85 instrument was used.

2.3 Scanning Electron Microscopy

JEOL JSM-5800 LV low vacuum scanning electron microscope was used for surface evaluation of a paper handsheet containing ISPCC prepared according to TAPPI testing standard T205 sp-06 [13].

3. RESULTS AND DISCUSSION

The production of ISPCC together with EC pulp fibers was carried out based on pre-testing of the LSLP system. For each ISPCC production run, two batches of 3 kg of OD EC fibers at a consistency of 4.5% were prepared using a 100 liter laboratory pulper. The resulting 133.3 l of pulp suspension containing 4.5% OD EC fibers. The resulting pulp fiber suspension is transferred into the LSLP system tank (1) and diluted to a consistency of 1.70% by adding to tank (1) 219 liter of H_2O to a final liquid suspension of 353 l.The $Ca(OH)_2$ suspension with 20% dry solids content containing 3kg (15 l), 6 kg (30 l), and 9 kg (45 l) based on the final targeted ISPCC level is prepared in the lime tank (8) prior to each ISPCC production run. After the $Ca(OH)_2$ suspension is added, mixing with a variable speed propeller mixer (3) is started by adjusting the mixer speed that a vortex indentation of approximately 100 mm (4 in) in tank (1) resulted. After mixing adjustment the recirculation pump (4) is started and the pulp suspension is recirculated at a flow rate of 64 l/min. the CO_2 addition is initiated by opening the pressure regulator of the Carbon dioxide (CO_2) storage tank (7) to a CO_2 gas flow of 60.0 cfm (1.7 l/min), this resulted into a pressure of 68948.0 Pa (10.0 psi) on pressure gauge (14), above the pressure reading on pressure gauge (15) located prior to the static mixer (6). The CO_2 gas exiting the CO_2 storage tank (7) transfers into a CO_2-heater (12) to maintain a CO_2 temperature between 20°C and 30°C to minimize ice buildup on the supply lines. The temperature of the CO_2 injected into the static mixer (6) is measured with temperature probe (13). The LSLP system is operated this way and temperature and pH values are monitored with probes (10) and (11) respectively. The LSLP system operation in stopped when pH of the recirculated pulp fiber suspension reaches a value of 7.0. The pulp fiber suspension with the ISPCC is then transferred into a receiving tank using transfer line (16) for further processing.

Fig. 2. Shows the ISPCC production run with 3 kg, 6kg, and 9 kg of $Ca(OH)_2$ added to the pulp suspension. After the 20% $Ca(OH)_2$ suspension was added the pH increased 12.77, 12.76 and 11.98 for the 3 kg, 6kg, and 9 kg respectively. For the following 30 min for the 3 kg and 9 kg $Ca(OH)_2$addition and 25 min for the 6kg addition the pH stayed above 12.0. A steep pH drop occurred in the following 10 minutes to a final pH of 7.29 and 7.28 for the 3 kg and 9 kg $Ca(OH)_2$ addition respectively and a pH of 7.55 for the 6 kg $Ca(OH)_2$ addition with final conversion of ISPCC achieved after 45 min. for the 3 kg and 9 kg $Ca(OH)_2$ addition respectively and 40 min. for the 6 kg $Ca(OH)_2$ addition. The pulp suspension temperature increased during in situ precipitation of ISPCC from 18.6°C to 20.8°C for the 3kg $Ca(OH)_2$ addition, from 20.2°C to 21.8°C for the 6kg $Ca(OH)_2$ addition, and from 21.1°C to 24.2°C for the 9kg $Ca(OH)_2$ addition.

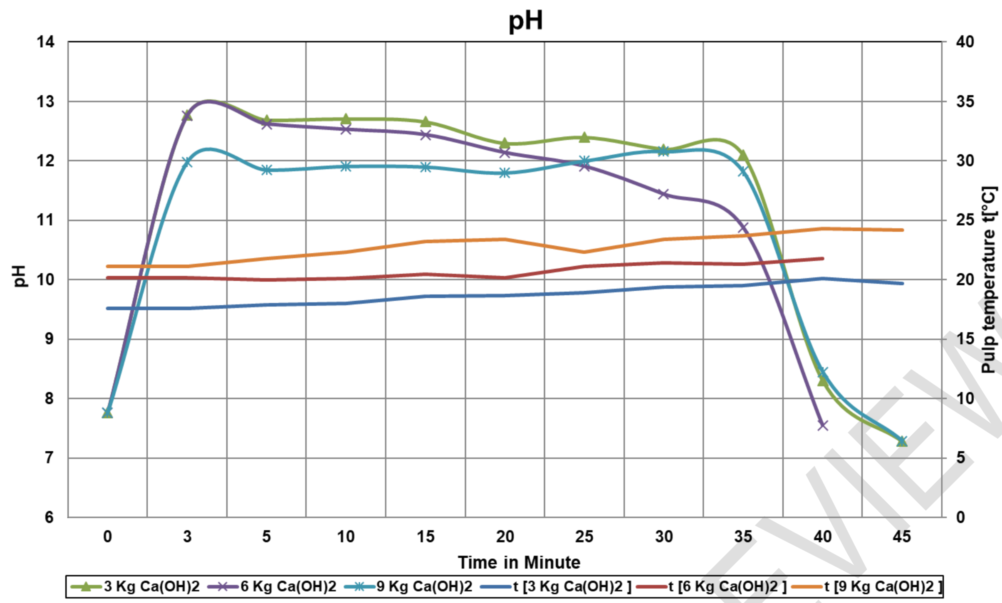

Fig. 2.: In Situ Precipitation of Calcium Carbonate

The ISPCC filler content based on OD pulp fiber suspension for the addition of 3 kg, 6 kg, and 9 kg $Ca(OH)_2$ addition was 38.2%, 55.5% and 66.6% respectively. The solids content after precipitation including EC fibers and ISPCC material was 1.80%, 3.52%, 4.38% for the 3 kg, 6 kg, and 9 kg $Ca(OH)_2$ addition respectively.

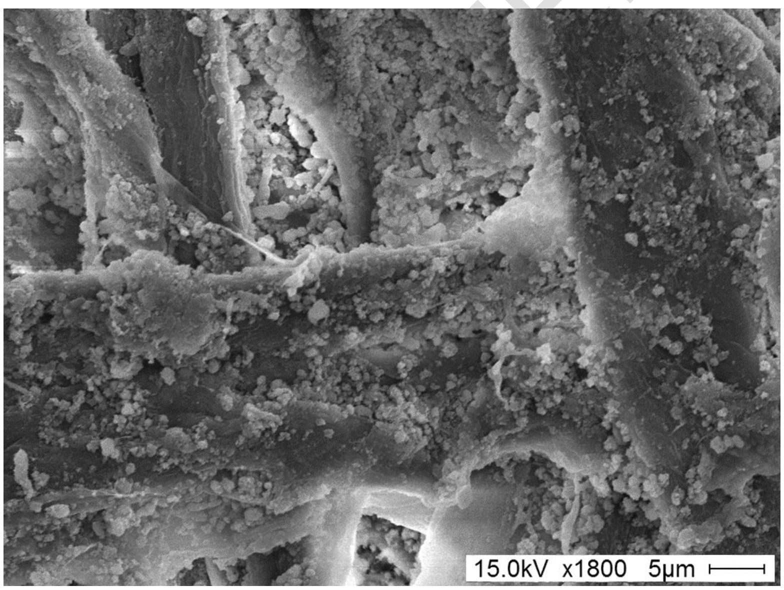

Fig. 3.: In Situ Precipitated Calcium Carbonate [16]

4. CONCLUSION

The development and design of a small pilot scale in situ laboratory calcium carbonate filler precipitation unit resulted a successful operation and production of a precipitated filler material in the presence of eucalyptus pulp fibers. Final filler content achieved was 38.2%, 55.5% and 66.6% based on 6 kg initial eucalyptus pulp fiber content. The precipitation time from an initial pH of 12.77, 12.76 and 11.98,a and final pH 7.29, 7.55, and 7.28 of for the 3 kg, 6kg, and 9 kg respectively was 45 minutes for the 3 kg and 9 kg $Ca(OH)_2$ addition and 40 min. for the 6 kg $Ca(OH)_2$ addition with a precipitation time.

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