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Production of Soft Ultrafine Functional Mineral Fillers with Super-Size Air Classifiers

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Abstract

Natural minerals like limestone and talc are used as fillers in plasticsand paints. These fillers trends in being more and more a funtional part of the final product.

The functionality of the fillers is mainly influenced from the particle size distribution. The finer the filler obviously the bigger the specific surface will be. With the classification technology state of the art new requirements cannot be reached. Therefore Messrs HOSOKAWA ALPINE developed a new classifier type realising the forced fortex.

In addition with the production of PCC also much finer grades down to 50%<0.05 micron.

We accept this challenge to achieve and even exceed the new requirements. Moreover there shall be found economical solutions to raise the profit.

HISTORICAL PERSPECTIVES OF FILLERS

In the last few years, production of mineral fillers has changed. Markets such as paper and plastics demand finer and finer products at higher capacities. Today, sometimes finenesses in the submicron range are required. This trend will finally lead to nanopowder particle sizes. Fillers have become more and more important in the industry. This is really a large step in the further development of Mineral Fillers.

When mineral fillers were first used in products, they were very crude and they had a very bad reputation. This is reflected in the statement.

"<u>Statement of a Boston Boot and Shoe manufacturers Assistant and Guide (1858)</u>

.... Manufacturers (of rubber articles) will tell you that fillers improve the article, bear greater pressure, etc., but as a rule this is mere trade subterfuge, the truth being it enable manufacturers to obtain extortionate profits

(printed in IM September 2000)

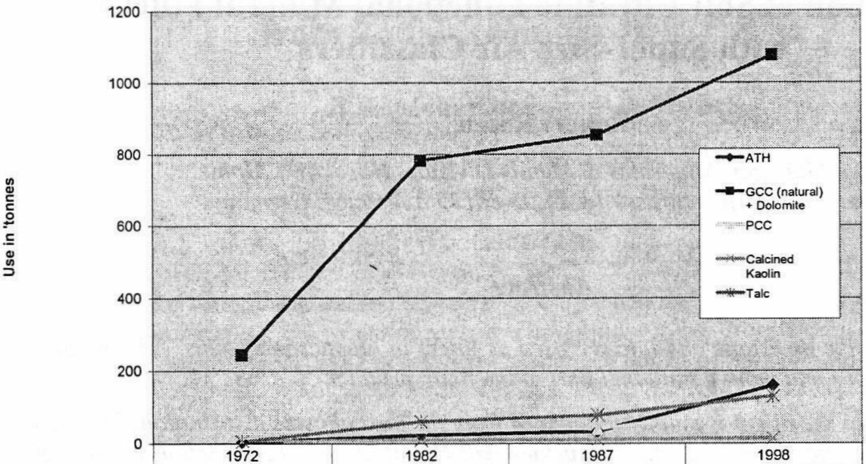
The real value of mineral fillers was neglected for a long time.

When the first oil crises hit the world economy in 1973, the raw material costs for polymer producers increased tremendously. Manufacturers were looking for cheaper raw materials in order to lower the costs of the polymer. This was the start for the further exploration of "mineral fillers".

The following chart (Source: Mineral Price Watch 02/2001) shows the development for fillers in Western Europe. There was a big capacity increase in usage from the years 1972 to 1982, especially for limestone.

By using "fillers", many properties of plastics such as those listed below can be manipulated.

Use of particulate fillers in Plastics in Western Europe ('000 tonnes)



	1972	1982	1987	1998
ATH	3.5	24	35	160
GCC (natural) + Dolomite	244	783	854	1076
PCC	30	46	42	100
Calcined Kaolin	8.3	10.5	12.5	15
Talc	6	62	80	131

Year

Tensile strength,

- Compression,
- Friction,
- Chemical resistance,
- Impact etc.

Filler became a real functional component of many products. In order to get away from the low cost image of previous years, the industry changed the name from fillers to

- Mineral additives
- Reinforcements
- Engineered fillers
- Functional fillers Etc.

With the higher importance of fillers, not only the name has been changed also the value. Fillers are not low-cost anymore. As a rule of thumb we can say that the finer the filler, the more expensive it will be. The particle size (Top size or d50-value) is sometimes only one important characteristic. There are other characteristics such as

- Density
- Particle shape
- Surface treatment
- Brightness Etc.

The systems in which fillers are mainly produced by dry processing are:

- Ball mills in circuit with classifiers
- Roller mills

- Classifier mills
- Impact mills in circuit with classifiers
- Jet mills

With the markets demand for fillers in the fine to ultrafine range as well as an increase in capacities, most existing system designs have achieved their limit. They just cannot produce the required fineness, or one system is not enough to produce the required capacity. In order to improve existing systems, companies are looking more and more for the addition of a classification step in order to

Ad a classifier into an existing system to post-classify the fines from the existing systems such as ball mill / classifying systems as well as roller mills

Replacing old classifiers in systems by more advanced ones

Retrofitting mills with different types of classifiers (e. g. Retrofitting of roller mills)

Classification has become more important in the last 5-6 years.

What is classification and how does a classifier work?

Classification is the separation of

- Smaller particles from larger ones
- Heavier particles from lighter ones
- Spherical particles from platy ones

by using a separation media, usually air.

Most existing classifiers incorporate a vertical or horizontal integrated classifier wheel. The wheel speed is adjustable by using a frequency inverter. Classification is a function of the peripheral speed or the speed of the classifying wheel and the radial speed of the air flowing through the classifying wheel, i. e. the mass flow rate of the air drawn through the classifier wheel by the fan.

The fines can be changed by setting the classifier wheel speed and air flow rates

Air flow	Wheel speed	Product	Capacity
Constant	Constant	Constant	Constant
High	Constant	Coarser	Higher
Low	Constant	Finer	Lower
Constant	High	Finer	Lower
Constant	Low	Coarser	Higher

Limitation of Traditional Single Wheel Classifiers

A. Capacity

With small diameter classifier wheels high finenesses with sharp topcuts can be achieved; however the capacities are very low. In order to achieve higher capacities several smaller classifiers have to be used. This leads to higher costs in

- Initial capital
- Operation
- Maintenance

B. High Fineness

With bigger classifier wheels, ultra fine fillers cannot be produced. There are mechanical design limitations with larger classifier wheels which prohibit the production of ultra fine products. Therefore, test results using small classifiers cannot be scaled-up to larger classifiers. Material fineness will suffer.

Solution

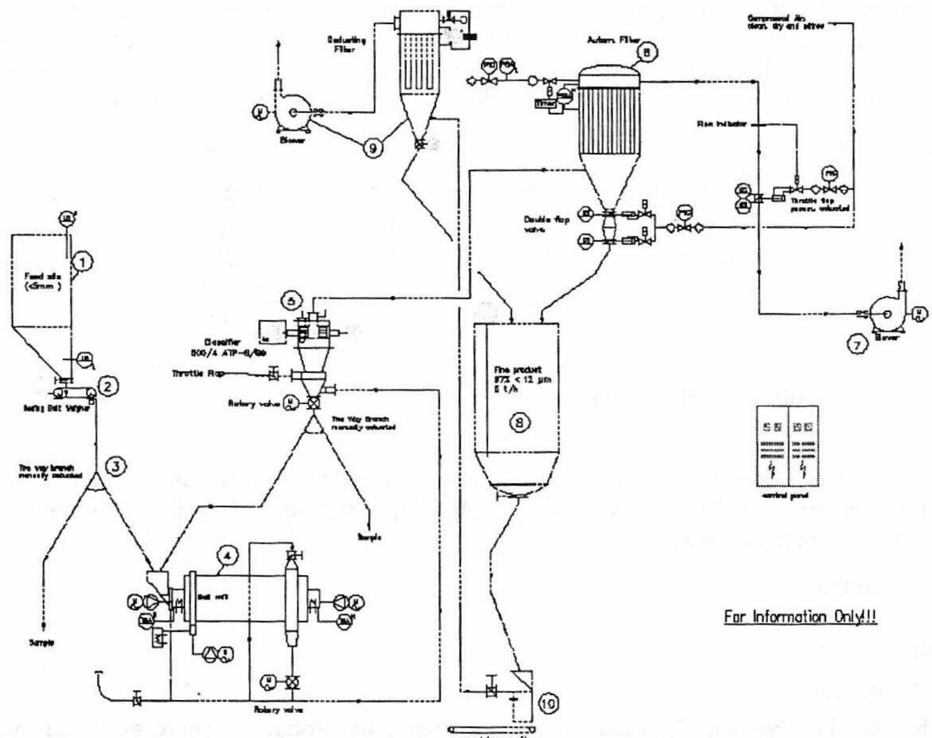
The solution to this dilemma is one classifier housing with several integrated smaller classifier wheels. With this patented Multi-Wheel-Classifier-Design, ultra fine fillers can be produced at high capacities. As an example, a classifier wheel with a wheel ∅ 315 mm, at maximum speed can produce a fineness of 99% < 6 microns at a capacity of 200 kg/h. With the multi wheel design 315/6, which has 6 classifiers wheels (each ∅ 315 mm), a capacity of 1,200 kg can be achieved. The sharp top cut is the same as the single wheel design. It is a single system with one feed inlet, one bag house and one blower.

The advantages of this system vs. several smaller systems are reduction in costs for :

- Initial capital
- Operation
- Maintenance

The following flow sheet shows a ball mill/classifying system for the production of an ultra fine limestone product.

The material is fed from a silo (1) and weigh belt feeder (2) into the ball mill (4). The dual flap valve (3) allows feed samples to be taken. The ball mill has a slot discharge design. The material exits the mill through slots in the grinding drum. Using the slotted discharge, the fineness of the material exiting the ball mill can be controlled. By closing more slots, the residence time in the mill can be increased. The result is a finer product. By opening more slots, the material exiting the ball mill will be coarser.



System, comprising of: Ball mill type 270/400 with classifier 500/4 ATP
System performance: $d_{97} = 10\mu m$ at 4,8 t/h

From the ball mill discharge, the material is pneumatically conveyed to the multi wheel classifier (5). The classifier is fed along with the main airflow. With this feed design, an additional feed device can be eliminated. The ultra fine material is collected in a bag house (6) whereas the coarse material is recycled by gravity feed back to the ball mill.

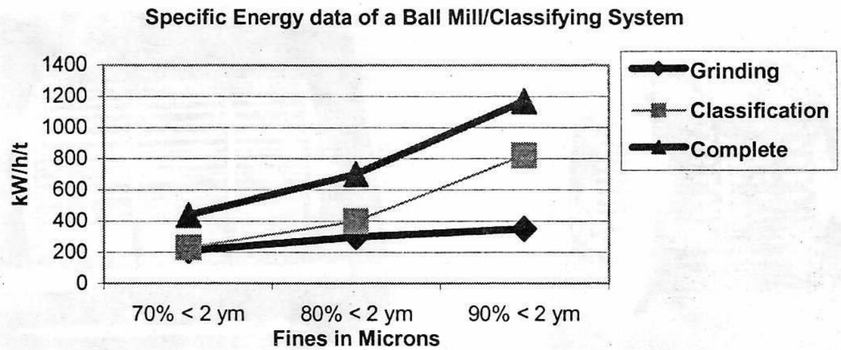
The blower (7) generates the classification air as well as the conveying for the material. From the bag house, material is fed into a storage silo (8) before it is bagged. The smaller bag house (9) with the blower serves as de-dusting station for the fines storage hopper as well as the bagging station (10).

Ball mill / Classifying System for Limestone

As mentioned above, classification has become more and more important. Unfortunately, with the production of ultra-fine minerals, the energy consumption of classifiers also increases.

The finer the product, the higher the energy input into the classifier (Drive power for the classifier wheels as well as the blower for generating the classification air). In the ultra-fine range, the classifier system can consume more energy than the grinder e.g. in a ball mill / classifying system.

The following chart shows the energy consumption for limestone processing for a vertical



Ball mill / classifying system at various finenesses.

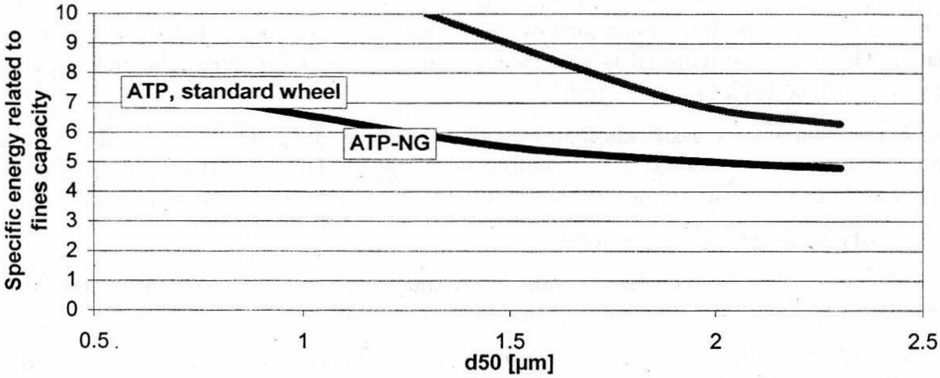
The finer the requirement for the material, the higher the classification energy will be. The ball mill energy consumption will be linear whereas the energy consumption, necessary for the classification air will develop exponentially. Since energy has become more expensive and therefore the products, everyone is looking for reduced costs in the way of energy savings.

There are no possible changes at the ball mill to reduce the energy consumption; however the classifier still offers a possibility for energy savings with its most important part; the classifier wheel. By changing the design geometry of the wheel, energy savings can be achieved.

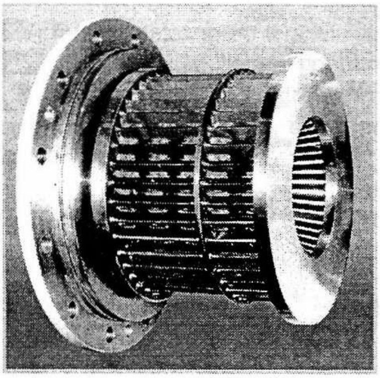
The following chart shows the energy consumption of a conventional wheel vs. the(NG) New Generation wheel.

The chart shows that the finer the product, the higher the energy savings. This is definitely one possibility to save energy as well as save capital costs due to the requirement for smaller blowers. The NG-wheel is also available for the multi wheel classifier design. This offers the possibility to produce ultra-fine fillers at higher capacities while consuming lower energy and capital costs.

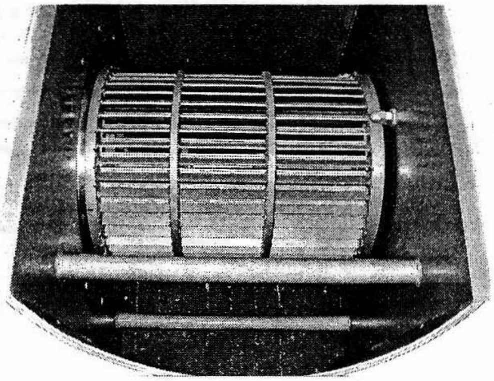
The multi-wheel classifier is still more expensive than a single wheel classifier. Since the mineral industry is always looking for cost reduction, an alternative to the multi-wheel design had to be found. The challenge was to design a classifier with the following properties



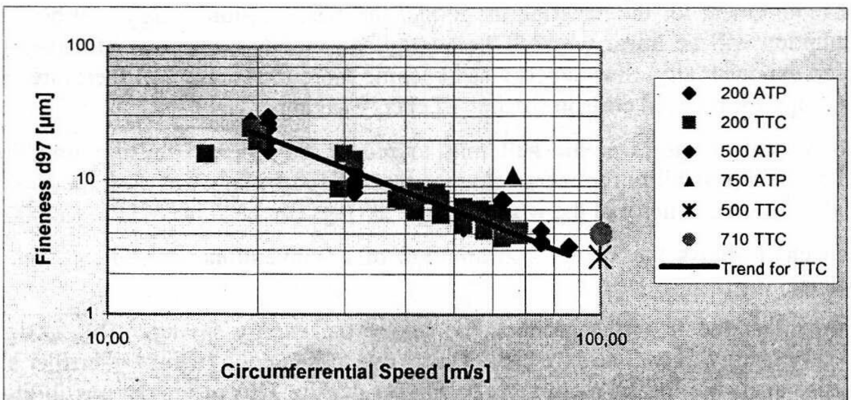
- Achieving the same finenesses as the NG-classifier
- Single wheel classifier
- Low energy consumption



NG-wheel



TTC-wheel



The solution was an extension of the NG wheel, running on 2 bearings. The new classifier "Turbotwin (TTC)" was borne. With its design, the above-mentioned properties could be achieved. Presently, the biggest available TTC is model # 710 with a max. installed classifier motor with 160 kW and an airflow of 20.000 m^3/h.

The above-mentioned table shows the comparison between fines and wheel speed between the standard ATP-wheel and the TTC wheel. It clearly shows that we are able to achieve much higher finenesses with the TTC wheel compared with a standard ATP-wheel with the same diameter. With the 500 ATP wheel, a fines of approx. 6-7μm can be achieved, whereas the 500 TTC wheel can reach 3μm.

Despite the development of the TTC, the big multi-wheel classifier such as 500/4 ATP (30.000 m^3/h airflow) and 630/4 ATP (48.000 m^3/h airflow), executed as standard ATP or NG wheel justify their existence for the production of ultrafine fillers at large capacities.

Today we are able to process ultrafine powders at large capacities with our ball mill classifier systems. The ball mill is a special designed mill, able to produce enough fines; the classifier is our multi-wheel head design 500/4 ATP-NG and 630/4 ATP NG

Parameters	**500/4 ATP-NG**	**630/4 ATP-NG**
Air volume in m^3/h	30.000	48.000
Max. wheel speed:	2800	2400
d_{97} = 5μm	1,4	N/A
d_{97} = 6μm	2,0	2,4
d_{97} = 8μm	3,2	4,8
d_{97} = 10μm	4,0	7,2

The latest trend in limestone processing is a combination of our supersized ultrafine classifiers such as 500/4 ATP-NG with a TTC classifier for post classification. The idea behind this set-up is the processing of ultrafine powders by post classification. The product, coming from the 500/4 ATP is already very fine with a top size of approx. D97=10μm.

Post Classification of limestone, coming from a ball mill/classifying circuit

The above-shown PSD curves are from a 500 TTC, being installed as post-classifier.

The feed material to the 500 TTC is already very fine. The final product with d_{98}=3μm is extremely fine. The TTC parameters were as follows:

Wheel diameter:	500 mm
Wheel speed:	4300 rpm
Air flow:	11500 m^3/h
Feed rate:	4,5 t/h
Fines capacity:	0,7 t/h
Fines yield	39,3%
Specific energy:	220 kWh/t

Comparing the feed curve and the curve of the coarse, one can see that the top size is the same; just the d_{50} is slightly different. This means that the coarse product can be sold as finished product as well as the fines. There is no by-product.

The fines with d_{98}μm has a very steep PSD with a clean top cut. It can be sold to a high price.

Since HOSOKAWA ALPINE Aktiengesellschaft also manufactures classifier mills besides the classifiers, the new developed TTC classifier wheel was also introduced to our jet classifier mills type AFG. This mill has been sold for many years for the most different applications in pharmaceutical, chemical and mineral industry.

By using the TTC classifier head instead the standard ATP head, the performance of this mill could also be improved. The new design is called TTG. The effects by using the TTC head vs. the ATP head are the same as for the classifiers.

- Higher capacity
- Higher fineness
- Lower energy consumption

The graph below shows the correlation between wheel speed vs. d_{50} value and the comparison between ATP and TTG. The product was talc. The graph clearly shows that the TTG wheel is able to make a finer product.

By using the TTC head on top of the AFG, the multi-wheel design can be replaced, resulting in lower capital costs. Especially for the soft minerals such as talc, wollastonite barite etc, the TTC head is a valuable option.

As mentioned earlier in this paper, the trend in mineral industry is more and more directed towards finer product. Today, in some areas finenesses in the range of 0.5 –1 μm (top size) are required. This is an area were traditional mechanical processing systems have reached their limitations. The usage of PCC has been known in the industry for many years.

As logical consequence of Alpine's philosophy to stay at the top of powder processing, the development of a PCC process was the next step.

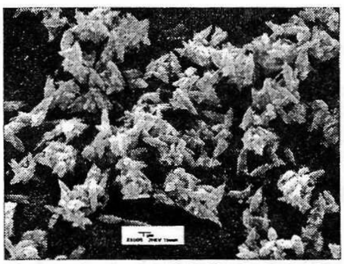
The specific advantages of PCC production are:

- Much finer products (down to 0,05 micron) at lower specific energy
- The specific energy for PCC is almost constant and not really a function of the fineness as operating costs for the production vary very little.

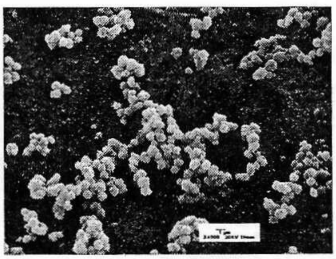
The resulting product can be controlled in many different ways by adjusting the parameters of the chemical reaction. The specific advantages of PCC products include

- Control of morphology or crystal shape
- Degree of aggregation or the clustering of the particles
- Particle size or specific surface area.

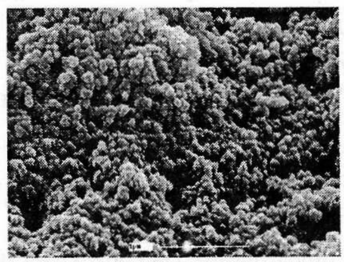
All these factors give us the possibility to design products for any given application.



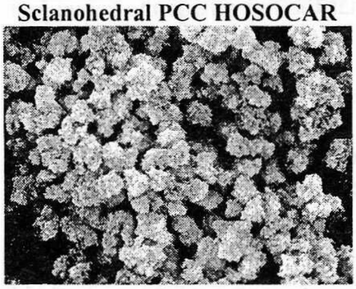
Sclanohedral PCC HOSOCAR



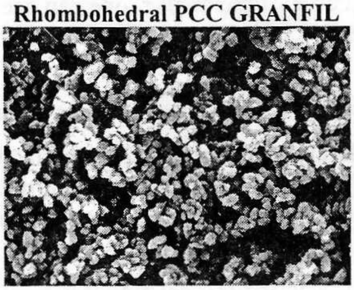
Rhombohedral PCC GRANFIL



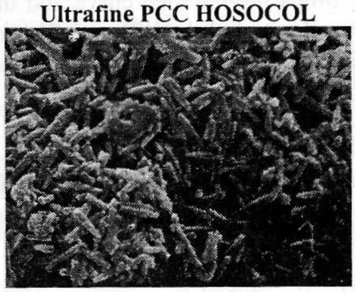
Ultrafine PCC HOSOCOL



Clustered Scalenohedral HOSOCLUST



Prismatic Calcite PCC HOSOFIL



Aragonite PCC

Fig. 1: HOSOKAWA Alpine's product range

Basic Morphologies

Over the last years HOSOKAWA Alpine has developed a variety of crystal shapes that can be used for many different applications, Picture 1 shows an overview of the product range. All these products except the aragonite PCC have been successfully produced on a large scale using our equipment and our know how. The aragonite PCC has still a R & D status but will be available in the near future.

THE PCC PRODUCTION PROCESS

Equations

The production of PCC consists of three steps as shown in equations (1)-(3). In the first step calcium carbonate is heated in a kiln up to a temperature of about 1050 °C, driving off CO_2-gas and producing calcium oxide (CaO), also known as quicklime. In a second step the slaking process takes place. In this process calcium hydroxide slurry is produced by mixing quicklime with a large surplus of water. In the final step the available CO_2-gas is reused and bubbled through the calcium hydroxide suspension, whereby PCC precipitates.

(1) Calcination: $CaCO_3 + \text{heat} \Rightarrow CaO + CO_2\uparrow$
(2) Slaking: $CaO + H_2O \Rightarrow Ca(OH)_2 + \text{heat}\uparrow$
(3) Carbonation: $Ca(OH)_2 + CO_2 \Rightarrow CaCO_3 + H_2O + \text{heat}\uparrow$

Flowsheet

Picture 2 shows a typical flowsheet of a PCC plant where all three chemical equations of the process are carried out. The process starts at the kiln which is fed with natural calcium carbonate. The waste gas containing the carbon dioxide has to be treated in two steps in order to be reused in the following PCC process. First the gas passes a filter which significantly reduces the dust load of the gas. Afterwards the gas passes a scrubber where the gas is cleaned from impurities and cooled down. The quicklime produced in the kiln is then fed into a slaker, where fresh water in a large surplus is added to the lime in order to produce the calcium hydroxide suspension. The suspension is then screened to avoid impurities or unreacted lime, also called dead lime. After this the slurry is cooled down and stored in a slake tank. The slake hold tank acts as a buffer between the continuously working slaker and the batchwise working reactors. In order to start a batch a reactor is filled with slurry from the slake hold tank. After the reactor is filled the cleaned waste gas is injected into the reactor until the reaction is finished. Depending on the specifications of the product we might have to control the temperature of the slurry in the reactor during the carbonation. Therefore we use a recirculation pump, which pumps the slurry through a heat exchanger back into the reactor. When the reaction has reached its final point the slurry is screened again, mainly to eliminate unreacted calcium hydroxide. After this the dewatering starts by pumping the slurry into the filterpress.

If powder PCC is required we need to feed the filtercake into an impact dryer leading to the final product. Normally end moistures of less than 0.5 % can be achieved. High solids slurry can be achieved by feeding the filtercake into a dispersion mixer.

Depending on the particle size the coating of the PCC can be done either in the dry or in the wet phase.

HOSOKAWA ALPINE'S CONCEPT

Quite in contrast to our competitors we don't sell the product itself but the equipment and the know how for its production. Our customer is operating the plant with the know how we deliver.

Plant Capacity

We can adapt the plant to the capacity needed by the customer. However, depending on the local costs for energy, crudes and man power a minimum size for the plant of 8.000-10.000 t/y is required. We can assist our customers at an early stage in making a feasibility study for the project.

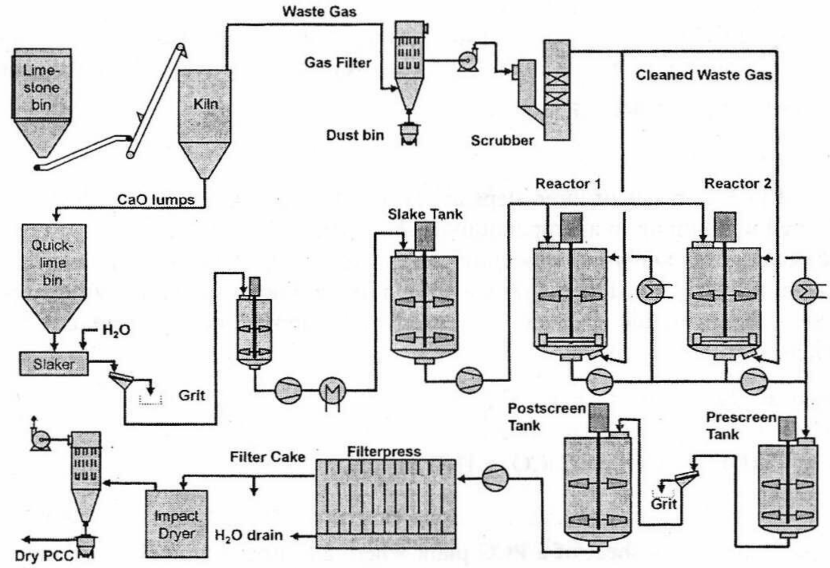


Fig. 2: Flowsheet of a PCC Plant

Applications, Product Specifications & Markets

There are many different industries that rely on PCC at some point of their production process. The focus however will be on the four most important industries: paper, plastic, adhesive and sealants as well as the paint industry.

Paper Industry

Originally mineral fillers were used in the paper industry in order to replace the very expensive fibres and to improve the printing properties of the paper. Today, minerals like Kaolin, Talc, GCC and PCC account for more than 90 % of fillers used in paper.

The paper industry is by far the biggest consumer of PCC using 73 % of the world-wide production of PCC. PCC is used in two different applications: paper filling and paper coating. The major use of PCC is the filling of woodfree uncoated paper (WFU) where the filler loadings can be up to 25 %. The final products are office and writing papers, magazines, books or advertising materials. But PCC is also used in supercalendered (SC) papers which are expected to increase the demand for PCC. SC paper is moving away from the newsprint quality and will compete more and more with light weight coated (LWC) paper. This will demand higher PCC usage in SC papers. Some papers need to be coated for some applications and printing methods. For this purpose coating pigments are spread as a thin film over the filled paper sheet. This film can be made extremely smooth by a combination of careful application and subsequent calendering steps. Originally 100 % kaolin was used for paper coating. Due to better whiteness, higher opacity and generally lower cost, PCC and GCC started replacing kaolin in this application. For the coating of paper PCC is normally blended with other minerals such as kaolin. Usually 45% - 95% of PCC is added to the coating formula.

HOSOKAWA Alpine has developed several products for the usage in the paper industry. Table 1 gives an overview of the products recommended for use in the papermaking process. However the products can be adapted to the needs of any customer.

Table 1: PCC for the use in Paper

Paper Filling		
	APS [micron]	**BET [m^2/g]**
HOSOCAR S1	0,9 – 1,3	11 – 16
HOSOCAR S2	1,9 – 2,2	5 – 7
HOSOCLUST	1,5 – 3,5	2 – 6
HOSOFIL S1	0,8	7
Paper Coating		
	APS [micron]	**BET [m^2/g]**
HOSOFIL S2	0,9 – 1,3	11 – 16
HOSOFIL S3	1,9 – 2,2	5 – 7
GRANFIL	1,5 – 3,5	2 – 6

HOSOCAR type filler provides an increased paper opacity and brightness and can be used in WFU or SC papers. While these products increase the bulk in the paper they also reduce the abrasion which leads to a longer lifetime of the felts and forming fabric. HOSOCLUST is a special product that might be used for cigarette paper or as a filler in WFU papers. It increases the paper strength and stiffness compared to the standard HOSOCAR products.

For paper coating HOSOFIL and GRANFIL can be used. These products provide a better ink holdout property, while the paper brightness and opacity can be improved at similar gloss rates.

Plastic Industry

Calcium Carbonate is by far the most widely used filler in the plastic industry, PVC (hard and soft PVC) being the most common application. PVC is a polymer used in a wide range of applications like window profiles, pipes, cables and fittings.

PCC is used as a filler in order to extend and cheapen the resin, but also as a non-reinforcing filler due to its positive effect on the performance of the plastic resin. Among these positive effects are :

- Improved impact strength, especially at low temperatures
- Higher gloss and improved surface finish
- Shorter gelation time

HOSOKAWA Alpine offers HOSOCOL as a filler in rigid and soft PVC. HOSOCOL is a colloidal product with a wide range of fineness (APS = 0,04 – 0,08 micron) for this application. The product might be surface treated depending on the application of the final user. Usually fatty acid is used for the surface treatment of the colloidal PCC.

Adhesive & Sealant Industry

PCC is used mainly in the production of expensive materials for the construction, electronics and aerospace market. Typical products in which PCC is used are PVC plastisols, silicon and PU sealants. Usually coated PCC is used for this task. PCC improves the working properties as well as the permanence or strength of the adhesive. It also can be used to modify the thermal expansion and conductivity. PCC used as an extender allows to reduce the amount of binder needed, thus reducing the production costs of adhesives and sealants. However, in some applications high filler loadings can have a negative effect on some properties of the adhesive. Therefore producers have to balance costs with performance in order to achieve the necessary product properties at acceptable prices.

HOSOKAWA Alpine recommends HOSOCOL as a filler for the use in high end application. HOSOCOL can be produced within a wide range of fineness and is surface treated for this application. Usually different kinds of fatty acids might be used for the coating of the colloidal PCC. Coated

HOSOCOL is aimed at the use in construction and automotive sealants. This includes PVC plastisols, polyurethane and silicones.

Paint Industry

In the paint industry PCC is used as a pigment. The main reason for the use of PCC is to replace expensive pigments such as titanium dioxide. Savings in titanium dioxide are claimed to be in the range of 10% to 35%. Furthermore PCC is used to increase the brightness and opacity of the paint as well as to control the rheological properties of the final product.

HOSOKAWA Alpine offers HOSOCAR as a filler in paint and coating applications. A typical HOSOCAR product for this application has an average particle size of 1,3 micron and a specific surface of 11 m^2/g. It is produced as a dry powder with an end moisture of less than 0,5 %. Typical characteristics of this product are its high purity and brightness. HOSOCAR can be used as a TiO_2 extender that improves the rheological properties of the final product.

ANNEX I

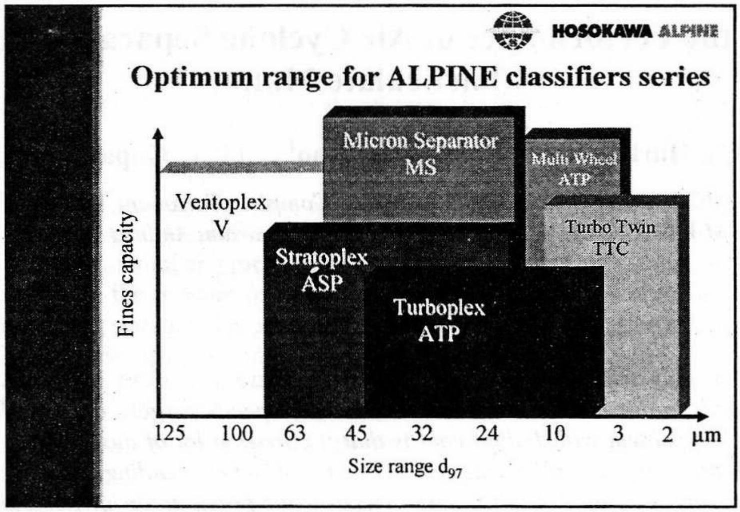
Particle Size Ranges for Different Applications

Applications	Size category (µm)	Mean particle size
Joint cement, carpet backings, asphalt roofing	coarse	22-50
Caulk, putties, sealants, rubber	medium	10-22
Paint, plastics, paper, rubber	fine	3-10
Paper, paint, plastics	ultra-fine	0.5-2

Physical Properties of Calcium Carbonate Fillers

	PCC (ultra-fine)	**GCC (ultrafine ground)**	**GCC (ground)**
Specific gravity	2.71-2.93	2.71	2.71
Index of refraction, mean	1.58-1.63	1.58	1.58
Hardness (Mohs' scale)	3-3.5	3	3
Decomposition temp.°C	800-900	800-900	800-900
Valley abrasion, mg	5-8	10	25
GE brightness, %	98-99	96	95
Oil absorption, $cm^3/100g$	30-55	23	13
Surface area, m^2/g	20-30 [1)]	9.6	3.2
The above-mentioned data are from the IM-magazine			
Has been changed by HOSOKAWA Alpine;			

ANNEX II



ANNEX III