emtec ACA Ash Content Analyzer

Measurement of the mineral filler content in paper and board

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Minerals in paper and the necessity of their measurement

Fillers are added to the pulp suspension to achieve certain properties. Minerals for example can have a positive influence to the degree of whiteness (intensity of white), opacity (light resistance) and to the flatness/printability of the paper. Additionally, because of the lower price – compared to primary fibers and partly waste paper – the use of minerals enables the reduction of costs for raw material.

Typical fillers in the paper industry are

Clay	With positive influence on the smoothness, gloss, printability. Used especially at wood-containing lightweight coated (LWC) paper and SC super calendared (SC) paper.
Calcium carbonate (CaCO3	As coating color pigment and filler, especially for the enhancement of the degree of whiteness of paper. It is comparatively low-priced. Precipitated calcium carbonate is required for special papers like cigarette paper.
Titanium dioxide (TiO2)	With very high degree of whiteness und high opacity. It is applied in large quantities in special papers like decor, label and banknote paper, and it is the most expensive pigment.
Talcum	Especially for the smoothing of the surface of graphic papers, for the usage in coating colors or for pitch control. It is applied in large quantities in LWC rotogravure paper and wood-free coated qualities.
Iron oxide	Applied as colored pigment (colors: yellow, red, brown, and black) in décor and tipping paper.

A great number of graphic and many other papers contain already a high percentage of minerals. Uncoated papers can be composed of up to 40 % by weight of minerals, similar to the amount in coated papers, whose surfaces contain coating pigments for a better printability. Fillers play an increasingly important role in the paper production and production cycle, which is also due to the enlarged usage of waste paper. The waste paper leads to an automatic supply of minerals in the production process. This represents a problem, because the single components of minerals and their amounts are unknown. The type of contained minerals strongly affects the characteristics of the finished paper. As a result, there is a need for permanent and accurate monitoring and control of the mineral content in paper/board and its influence to the process. The total content as well as the single components and their percentage distribution must be known, because, for example, the different components can have a different retention, and for the optimization, the respective retention agents should

be applied. Another necessity is a quick supply of this data, as the production speed is usually very high. This information about the mineral content should be gathered at different points of the production chain, because the concentration of minerals in the waste paper and in the final product does change with the addition of further minerals, and can also change technologically conditioned.

Previous methods for the measurement of the filler content

Traditionally in the paper industry, the ash content or the combustion residue is determined by the combustion method according to ISO 1762 and 2144, DIN 54370, TAPPI T 413 or 211.

1. Determination of the ash content by combustion at 525°C ± 25°C

   At first, the sample is combusted (until the residue does not have carbon from the organic material). All inorganic components, meaning mineral fillers and coating pigments, of the sample remain, as mineral components are incombustible. In uncoated paper the fillers mainly remain, while in coated paper also the coating pigments.

2. Determination of the combustion residue at 900°C ± 25°C

   Afterwards, the glow of the residue takes place (which is necessary for the determination of the calcium carbonate). The sample is combusted directly at 900°C ± 25 °C, if it does not contain calcium carbonate.

Disadvantages of the traditional combustion method

Figure 1: Combustion method

Although it is possible to determine the total content, only the content of few selected fillers (calcium carbonate, titanium dioxide + clay) can be measured. A further disadvantage of the method is the glowing loss, which is the reason why the ash content cannot be equalized with the filler content. The extend of the glowing loss differs for different fillers and coating pigments. The ash content measurement varies from company to company, depending on the used standards (e.g. company-internal standards) and combustion temperatures (e.g. 525°C and 900°C vs. 575°C and 925°C), the picture illustrates this. The combustion of the samples is user-dependent. To avoid random or methodical mistakes, it is important to work very accurately and thoroughly. Although under optimal conditions, it is assumed that the accuracy of the combustion method is approx. ± 2 %, a relatively inaccurate combustion of the samples

of up to ± 5 % arises in routine operation. Often the quick combustion is used, which takes place 10 – 20 minutes at 900°C (depending on the type of paper), whereby clay cannot be determined, if calcium carbonate is also in the mix. The heating of the oven is skipped partly. The measurement turns to be very time-consuming (approx. 3 – 7 hours in total), especially when requiring high accuracy in accordance with the ISO standards. A non-optimal process control as well as an over dosage are caused by the delayed availability of measuring results, because wide specifications become necessary. Furthermore, it is disadvantageous that the combustion is a destructive measuring method. Thus, the traditional combustion method has numerous deficiencies. This created the need to develop a new measuring method for the determination of filler content. For this purpose, emtec Electronic developed the ACA Ash Content Analyzer, which will be described in the following.

Determination of mineral filler content using the ACA Ash Content Analyzer

Emtec's ACA Ash Content Analyzer and its new and innovative measuring principle without combustion of the paper samples, meaning without destruction, enable to determine both within seconds, the total filler content as well as the percentage content of typical fillers in the paper industry. This means that the respective percentage content of calcium carbonate, kaolin, talcum, and titanium dioxide as well as further used mineral fillers in the paper industry can be detected.

Measuring principle of the ACA

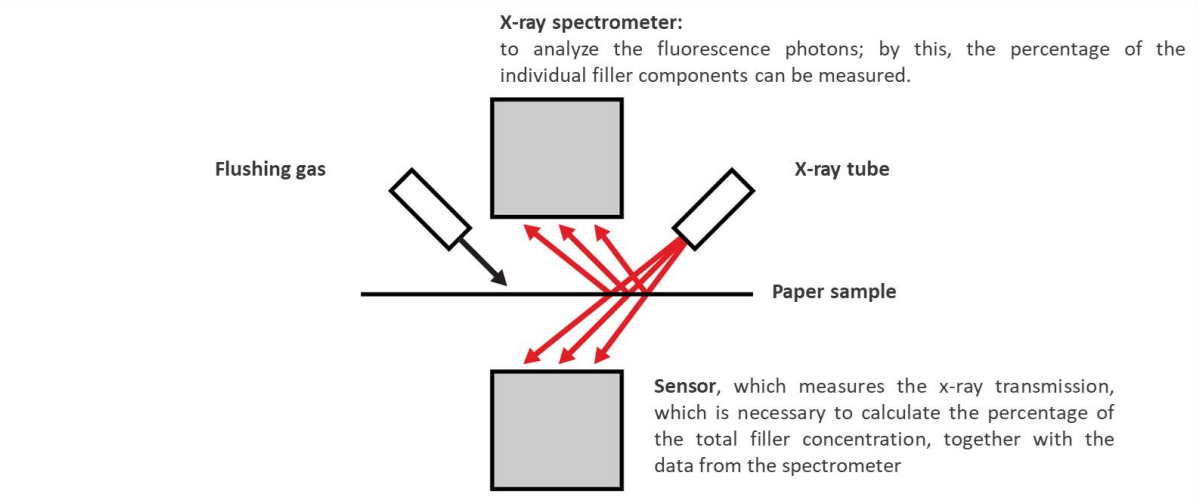
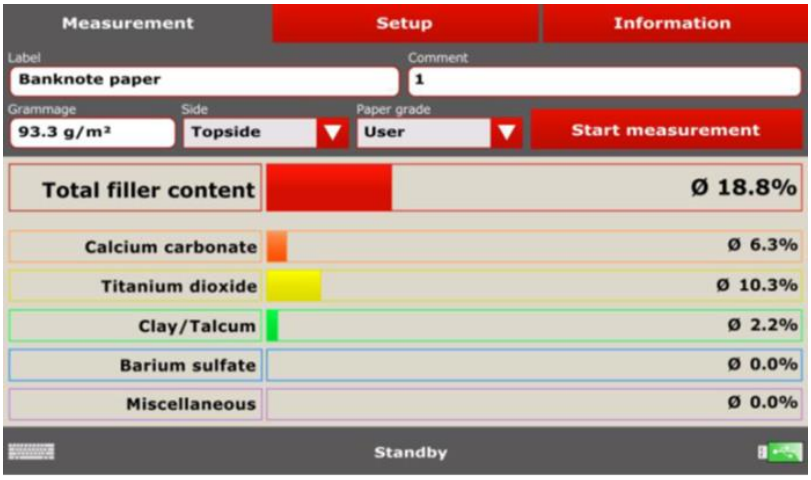


Figure 2: ACA measuring principle

The measuring method of the ACA bases on the combination of X-ray fluorescence analysis and the transmission method. First, the captured X-ray fluorescence spectra are qualitatively

evaluated, and afterwards, they are quantitatively determined concerning the concentration of the detected filler components. The signal peaks are converted using complex mathematical functions (algorithms) in the corresponding concentrations. Figure 2 illustrates the measuring principle.

Data analysis with the ACA



Display of results: mineral filler content of a paper sample (example)

Figure 3: Display of the ACA test results

The ACA is characterized by an especially simple handling. Label, grammage and optionally a comment can be entered, the measurement is started, and after about 45 seconds the total filler content and the percentage of each mineral filler, for example, calcium carbonate, titanium dioxide, kaolin, talcum and barium white are displayed. The following example of decor paper samples, measured with ACA and combusted at 525°C und 900°C, shows the correlation to the combustion method:

	Combustion		ACA test results			
Label	**525°C**	**900°C**	**Total filler content (%)**	**$CaCO_3$ (%)**	**TiO_2 (%)**	**Kalium/Talcum (%)**
1	31.7	31.6	31.2	-	31.2	-
2	27.1	26.5	27.5	-	19.8	7.7
3	30.1	29.9	29.7	-	29.7	-
4	12.2	12.1	12.4	-	12.4	-

Comparison measurements with ACA and combustion method

Figure 4: ACA vs. combustion method

Calculation of the results

There are two different types of calculations:

1. Global calibration

   With this, all samples (uncoated paper/board) can be measured. The data evaluation is based on the physical theory; the accuracy is material-dependent limited: up to approx. ± 0.5 - 2% (abs.).

2. Grade-/type-specific calibration

   By using of representative reference samples for each grade or type of similar paper with exactly known filler content (determination via combustion or TGM

   [thermogravimetric measuring procedure]) for calibration, the accuracy can be increased significantly: up to approx. ± 0.1 – 0.3 % (abs.).

Benefits and advantages of the ACA meassuring method

Features of the ACA

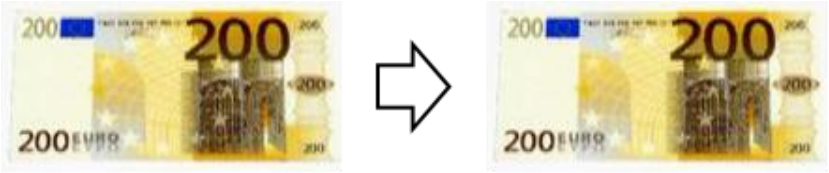
» Both, total content and the content of the typical fillers in the paper industry, can be separately identified and quantitatively determined

» Quick availability of measuring results (immediately respectively within seconds)

» High measuring accuracy: approx. ± 0.1 – 0.3 % (abs.) for grade-specific calibration, ≤ ±0.5 – 2% (abs.) for global calibration

» Low dependency of the results on the moisture of the sample

» Nondestructive measuring method



Before measurement After measurement

» Easy handling (self-explanatory operation)

» Easy portable (in trolley)

- Efficient calibration of online ash sensors possible with the ACA (especially useful in case of frequent grade changes!)

Hence, the ACA replaces the time-consuming traditional combustion methods. Furthermore, it offers significant advantages and delivers information, which is not possible to achieve by combustion.

Advantages for the user

- Efficient process optimization: Improve of filler retention
- Significant savings of working time by rapid determination of the cross profile of the filler distribution
- Optimal process control through prompt measurement results
- Monitoring the filler development along the entire production process, from raw material input to the finished product. Samples for measurement can e.g. be taken at the dump chest, at the input and output of flotation, bleaching tower, headbox and paper from PM (Suspensions can be examined as follows: creation of sheets on filter paper using the so-called “Nutsche” [“Büchner funnel”] or a sheet former, drying, and measurement with ACA.)
- Fast and targeted influence on the filler content respectively de facto instant response to changes in the process
- Reduction of process fluctuations
- Targeted control of the filler composition and content along the entire production chain (filler content in waste paper, before / after flotation)
- Faster attainment of the target values of the individual filler components during grade changes
- Targeted, effective product and quality development

Immense saving potential

- Savings in time, personnel, and energy compared to the combustion
- Substantial material savings by avoidance of low qualities and rejects
- Reduction of the consumption of pigments, as the greater accuracy of the ACA and the rapid availability of results allow for a **narrowing down of the specifications** for the filler content in the finished product (for process optimization)

Application areas

- » Décor paper
- » White top liner
- » Cigarette paper
- » Copy paper
- » Silicone paper
- » Any printing paper
- » Hand sheets / Buechner-Fannnel sheets
- » Retention trials
- » Recovery of fillers from the sludge treatment
- » one side coated board
- » Banknote and security paper

Application examples/return on investment

Example décor paper production

Production:	100,000 t / year
Consumption of titanium dioxide:	40 % = 40,000 t = 40,000,000 kg
Price of titanium dioxide:	1 kg = approx. 3 €
+ Transportation costs:	1 t = approx. 50 € → 1 kg = 0.05 €
= Price per kg	1 kg = 3.05 €
Costs per year:	approx. 122,000,000 €

Combustion method	Accuracy in routine operation approx. ± 5 %, i.e. specification must be relatively broad, i.e. 40% ± 5%.
ACA	Accuracy approx. ± 2 %, i.e. specifications can be 40 % ± 2 %.

⇨ **Improvement in accuracy** 3%-points

This results in a direct savings potential of 3,660,000 € (!) in best case

⇨ Only considering these direct costs leads to a ROI of only approx. 9 days (!)

Example copy-paper production

Production:	300,000 t/ year
Consumption of calcium carbonate:	20 % = 60,000 t = 60,000,000 kg
Price of calcium carbonate:	1 t = approx. 50-150 € → 1 kg = 0.10 €
+ Transportation costs:	1 t = approx. 50 € → 1 kg = 0.05 €
= Price per kg	1 kg = 0.15 €
Costs per year:	approx. 9,000,000 €

A 3% saving compared to combustion method results in a savings potential of 270,000 €
(2 %: 180,000 €) → Only considering these direct costs leads to a ROI of approx. 4 months (!).

Hint:

Although indirect savings such as the reduction of complaints cannot be specified herein, they should still be considered. Of course, the prices for transportation costs do vary from region to region. These are just mean values. The numbers can be adjusted to own conditions.

Technical data

Measurable mineral fillers	Calcium carbonate, Kaolin, Talcum, Titanium dioxide, Barium white, iron oxide
Accuracy of measurement	up to approx. ≤ ± 0.1 – 0.3% (abs.) for grade-specific calibration, approx. ≤ ±0.5 – 2% (abs.) for global calibration
Measurement time	approx. 45s, simultaneous recording of ambient temperature and humidity at each measurement
Grammage	maximum approx. 1000 g/m2
Dimension	approx. 315 x 400 x 280 mm (width x height x depth), depth with opened display: approx. 415 mm
Weight	approx. 14 kg
Power supply	100 - 240 VAC (50/60 Hz)
Data storage	Automatic storage of measurement data internally respectively on the plugged-in USB flash drive
	The device control and the measured value display can be done via the touch display of the device or, even more

comfortable, via PC with the emtec Measurement System Software including the data storage and special evaluation

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