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On the Mechanism of Sizing with Alkyl Ketene Dimer (AKD): Physico-chemical Aspects of AKD Retention and Sizing Efficiency

by

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INTRODUCTION

In recent years paper production has undergone a sweeping change from acid to alkaline paper making. Important driving forces for papermakers to work in a pH range of 6 to 9 are the possible use of calcium carbonate as a relatively inexpensive and good quality filler, improved paper durability, reduced machinery corrosion and increased paper strength.

Changing to alkaline pH has forced also modifications in wet end sizing systems. The traditional rosin-alum sizing method used in acid systems is unsuitable in the alkaline environment. High pH leads to poor size retention and thus to a loss in sizing efficiency. Instead, synthetic sizing agents such as alkyl ketene dimer (AKD) or alkenyl succinic anhydride (ASA) are the choice.

AKD belongs to the class of reactive sizing agents, i.e. the AKD molecule reacts with the cellulose fibre surface thereby rendering the fibre surface hydrophobic. The degree of sizing is believed to depend on the extend of the chemical reaction taking place between the AKD molecule and reactive sites on the fibre surface (Figure 1). Sizing is not immediately developed as with rosin-alum based systems, where the anchoring of rosin to the cellulose surface is achieved by electrostatic interactions [1;2].



Figure 1: Covalent bonding of AKD to the cellulose fibres

In spite of the fact that AKD has been widely used in the paper industry for many years, the mechanism of AKD sizing is still a point of discussion. It is generally assumed in literature that sizing efficiency depends on at least the following factors:

- · retention / deposition of AKD particles on the cellulose fibre
- · distribution of AKD particles on the fibre surface and consecutive spreading
- extend of the chemical reaction with the fibre surface and orientation of the hydrophobic groups

Fundamental understanding of these factors is necessary in order to optimise the paper making process and to minimise the amount of size needed to achieve the desired sizing targets. Excessive usage of sizing agent is not only costly but also contributes to the contamination of white water systems and the formation of deposits causing runnability problems. The ongoing closure of production systems, the incorporation of higher levels of recycled fibres and new retention strategies are having a strong influence on sizing efficiency. Therefore better understanding of the sizing mechanism and the process of size development of AKD is required. The first step towards effective sizing is optimum retention of AKD particles. The mechanism of retention is believed to be heterocoagulation of the cationic particles to the negatively charged fibre surface [3]. In order to evaluate the effect of retention on sizing quantitatively, one has to be able to quantify the amount of retained AKD in the wet end of the paper making process. The extremely low concentration of AKD needed for sizing makes the measurement very difficult and tedious.

Several methods to quantify retention of AKD on the fibre surface have been described in the literature: isotopic labelling of AKD [4;5], capillary gas chromatography [6], extraction and ketone analysis [7-9], pyrolysis gas chromatography [10] and visible/UV-spectroscopy [11]. All of these methods are too complicated and costly for the conduction of investigations in a large multi-parameter field. Time consuming and complex preparation procedures, such as the extraction of AKD from paper sheets and subsequent chemical processing of the extract, are the source for experimental errors and inaccurate results.

Therefore we have developed a new, reliable method, which measures the concentration of non-retained AKD after filtration of the pulp suspension. This new method is based on the spectrometric evaluation of the filtrate after selective dyeing of the hydrophobic AKD particles with a proprietary fluorescent dye.

Using this new technique we are able to study the interaction between fibres, fillers, the AKD dispersion and other process chemicals and their influence on the retention of AKD particles. Furthermore we will compare the degree of retention to the actual degree of sizing, which has been developed for a given situation.

MATERIALS

AKD dispersions

For our investigations we used two different types of AKD dispersions (commercially available by BASF under the trade name Basoplast[®]), which have been stabilised either with cationic starch (type "A") or with a newly developed synthetic stabiliser (type "B"). Both types contain 12% AKD. The dispersions were prepared using a laboratory homogeniser. The volume average particle size distribution of both dispersions can be characterised as a sharp distribution function ranging between 0,8 and 1,2 μ m.

The charge characteristic of the dispersions as a function of pH is shown in Figure 2.



Figure 2: Electrophoretic mobility of dispersion "A" and "B"

In the case of dispersion "A" one can observe a slight decrease in electrophoretic mobility as the pH is increased. The mobility at pH = 8 is found to be 0,5 (μ m/s)/(V/cm) for "A". The mobility value determined for "B" is much higher and nearly constant in the pH-range between 5 and 9. At pH 8 the mobility value is about 3,0 (μ m/s)/(V/cm). A strong decrease of the electrophoretic mobility is observed at pH-values larger than 9. From dispersion is cationic and significantly lower than the surface charge density of type "B" - dispersions.

Fibres, fillers and process chemicals

Retention of AKD particles has been studied under two different conditions:

- In case of laboratory conditions bleached sulfite pulp was used as the only fibre source. As fillers, anionically modified grounded calcium carbonate (GCC) and rod-shaped precipitated calcium carbonate (PCC) was used in this study. As retention aid, we have studied the effectiveness of Polymin[®] SK, a high molecular weight polyethylenimine and Solvitose[®] 2N, a cationic cold water soluble starch.
- Under practical conditions (pilot paper machine at BASF in Ludwigshafen) different types of pulps were used together with fillers and retention aids:
 (1) combination of TMP and bleached sulfite pulp; Hydrocarb OG (GCC) as filler; Polymin[®] KE 78 (cationic polyacrylamide) as retention aid;
 (2) recycled paper; no filler, Polymin[®] KE 78 as retention aid.

EXPERIMENTAL

Retention measurements

In case of laboratory conditions 5ml AKD dispersion was added to 500ml pulp suspension under stirring in a standard laboratory jar. The solids content of the pulp consisting of fibres or a mixture of fibres, fillers and/or other process chemicals was 8g/l. The concentration of AKD in the dispersion was chosen such that the concentration of (pure) AKD in the pulp suspension was varied between 0mg/l and 20mg/l. This corresponds to an AKD content of 0 - 0.25 wt.% with respect to the solids content of the pulp suspension. After a contact time of 5 minutes the non-retained AKD particles have to be separated from the fibres to enable their concentration to be measured in the filtrate. For this purpose a dynamic drainage jar is used (Figure 3). Filtration of the pulp suspension was carried out through a screen with a mesh of 40um. The filtrate, which contains AKD particles, pitch particles, fillers and fines smaller than 40µm, is diluted as required, and a solution of fluorescent dye in ethanol is added. The mixture is then left to stand for 30min at elevated temperature under stirring. After this procedure, a small volume was taken from the solution and was transferred to the sample cell inside a fluorescence spectrometer (Hitachi F4010). Inside the spectrometer the emission spectrum is measured for excitation around the main excitation band.



Figure 3: Principle of the AKD retention measurements

The quantum efficiency and the emission wavelength of the fluorescent dye strongly depends on the polarity of the medium. Therefore, at the emission wavelength λ_{np} , which is typical for the dye in a hydrophobic medium, the fluorescence intensity is related to the total amount of dye taken up into the hydrophobic phase. If the amount of dye added to the solution is sufficiently high, one can observe a linear relationship between the concentration of AKD (present as dispersion particle in the aqueous medium) and the

fluorescence intensity measured at λ_{np} in the typical application range between 0mg/l and 20 mg/l AKD. This linear relation, which has been measured for a diluted AKD dispersion without other hydrophobic components and fibres, can be used as a calibration curve for retention measurements (I₀ in Figure 4).

For an actual retention measurement the fluorescence intensity at λ_{np} is taken from the filtrate sample for different concentrations of AKD added before the filtration step. The result of such a measurement (I_f) is shown in Figure 4. The background fluorescence intensity, due to the presence of pitch particles and other hydrophobic compounds, has been subtracted. The linear dependence of I_f indicates that the degree of retention is constant for the range of typical AKD addition to the pulp. The retention value R [%] is calculated from the ratio of slopes S_f and S₀ measured for I_f and I₀ respectively:

$$R[\%] = (1 - S_f / S_0) \times 100$$
(1)

where R is the calculated retention value and S is the slope of the data points for the pure dispersion in water (S_0) and the not retained dispersion in the filtrate (S_f) .

Under practical conditions (pilot paper machine) white water samples (first pass retention) were collected continuously and analysed in order to determine the amount of non-retained AKD particles as described above.

Electrophoretic Mobility

Electrophoretic mobility measurements were performed using a microelectrophoresis apparatus PEN KEM 3000. Therefore the AKD dispersions were diluted (1:3000 by weight) with deionized water containing 10 mmol/l potassium chloride. The pH-value was varied between 4 and 11 by addition of sodium hydroxide and hydrogen chloride.





Figure 4: Fluorescence intensity vs. AKD concentration

Ketone analysis

For the ketone analysis the paper sheets were cutted in small pieces. A weighted part of the paper was taken and extracted twice with water free chloroform. The organic phases were combined and the solvent was evaporated. The residue was dissolved in a defined volume chloroform, containing the internal standard. An aliquot of the solution was taken and subjected to capillary gas chromatography. Only the peaks of the three most dominant ketones (bis-palmitylketone; palmityl-stearylketone and bis-stearylketone) were used for the quantification of the amount of ketone in paper.

Degree of sizing

The degree of sizing was evaluated by determination of Cobb value (according to DIN 2.08.03 or Tappi standard T 441 OS-63) and ink penetration time (according to DIN 2.08.04).

RESULTS

Two-component system: Fibre - AKD dispersion (laboratory conditions)

In the first step we studied the interaction between AKD particles and cellulose fibres in the two component system at pH 8. In case of type "A"-dispersion the degree of retention was found to be around 10%. These values are in good agreement with those found by Lindström et al. [4] using ¹⁴C-labelled AKD. In case of type "B"-dispersions non-retained AKD could not be detected in the filtrate, indicating 100% retention on the fibres.

type of dispersion	stabiliser	electrophoretic mobility (µm/s)/(V/cm)	retention value [%]
"A"	starch	0,5	10
"В"	synthetic polymer	3,0	100

Table 1: electrophoretic mobility and retention value of dispersion "A" and "B"

The observed difference in affinity for type "B"-dispersions compared to type "A"dispersions correlates with the difference in electrophoretic mobility measured for both dispersions at pH 8 (table 1).

The electrophoretic mobility of starch-stabilised dispersions can be increased by various methods such as the direct chemical modification of starch (degree of substitution with

cationic groups) or the formulation with cationic polymers. Further measurements, using such modifications, verify the strong correlation between mobility and retention value.

Using only synthetic cationic polymers as stabilisers for AKD the electrophoretic mobility is shifted in the range of 2,5 to 4 (μ m/s)/(V/cm) leading to quantitative retention. However, 100% retention can be already achieved with mobility values around 2,0 (μ m/s)/(V/cm) (Figure 5).



Figure 5: Retention value vs. electrophoretic mobility of AKD dispersions in a two component system

Influence of retention aids

Retention aids are mainly applied to improve the retention of fines and fillers and to accelerate the dewatering process. Additives used for this purpose are often neutral or charged polymers which have a certain affinity to both, the fibre and the component to be retained. Although the surface charge of our AKD dispersion particles is cationic, the influence of cationic retention aids on their retention behaviour is significant. The interaction of two different retention aids with dispersion "A" was investigated: Polymin[®] SK, a high molecular weight polyethylenimine and Solvitose[®] 2N, a cationic cold water soluble starch.

Adding constant amounts of Polymin[®] SK to the pulp, the degree of retention for dispersion "A" was determined. Addition of 0,05 wt.% Polymin[®] SK already leads to a significant increase of retention, while almost quantitative retention is reached at 0.1 wt.% Polymin[®] SK (Figure 6).

Compared to Polymin[®] SK the cationic starch Solvitose[®] 2N is rather ineffective with respect to an improvement of the retention levels. With the addition of 0,45% starch a retention level of about 70% is reached; for quantitative retention about 2% of Solvitose[®] 2N is necessary (Figure 6).

No significant change of the electrophoretic mobility of AKD dispersion particles was observed with the addition of either Polymin[®] SK or cationic starch Solvitose[®] 2N. Therefore we conclude, that the improvement of retention is not caused by direct adsorption of retention aids on the dispersion particles but rather by the adsorption on fines and fibres, leading to coflocculation of fines, fibres and AKD particles.



dispersion (1,25mg AKD/g fibre)

Interaction of AKD dispersions with fillers

In paper making fillers are used to reduce the cost of raw materials and to improve paper properties like opacity and printing performance. It is also well-known that a large number of additives and process chemicals used in the wet-end are readily adsorbed on the filler due to their high surface area. In case of AKD dispersions this is already well-documented in the literature [1; 12-15].

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The adsorption isotherms for type "A" dispersion particles on anionically modified ground calcium carbonate (GCC) and rod-shaped precipitated calcium carbonate (PCC) are displayed in Figure 7.



Figure 7: Adsorption isotherms of dispersion "A" on fillers (GCC; PCC)

	electrophoretic mobility (µm/s)/(V/cm)	BET surface area (m ² /g)
rod-shaped PCC	+ 2,0	6,0
anionically modified GCC	- 2,6	6,5

Table 2: characterisation of the fillers GCC and PCC

As one can see from the data in Figure 7 and table 2, in the practical range of addition (1 - 5 mg pure AKD/1g filler), the sign of the electrophoretic mobility value does not have a large effect on the affinity of type "A" dispersion particles. The degree of retention varies between 100% (GCC) and 80% (PCC). A similar result was also found for type "B" dispersion (90% retention for GCC and PCC).

The effect of filler content (anionically modified GCC) on the retention level of type "A" dispersion in a three component system (pulp/filler/AKD) is displayed in Figure 8. As one can see the retention level increases from 10% up to 60% when the filler content is raised from 0 wt% to 12.5 wt.%. Further increase of the filler content does not lead to a higher AKD retention level. This result can be understood, if one also takes into account the retention of fillers in the fibre web. As the adsorption of AKD particles on anionically modified GCC is quantitative, a certain part of the dispersion particles will always be attached to the non-retained filler particles and thus will be found in the filtrate.





Figure 8: Influence of filler content on the retention value (dispersion "A"; 1,25mg AKD/g fibre)

Four-component system pulp/AKD dispersion/retention aid/filler

In case of four component systems containing cellulose fibres, filler, retention aid and AKD dispersion, one would expect the best result for AKD retention, using a combination of fillers and retention aids. Laboratory experiments were performed with a system containing 87,5 wt.% bleached sulfite pulp and 12,5 wt.% anionically modified GCC. As a retention aid, Polymin[®] SK was added at different doses up to 0,15 wt.%. The result is

shown in Figure 9. Addition of Polymin[®] SK to the three component system only leads to a slow increase of AKD retention. A retention level of about 80% is reached at a Polymin[®] SK dose of 0,15 wt.%. In comparison with the GCC-free system the same retention value is already reached at a significantly lower addition level (0,08 wt.%).



Figure 9: Effect of Polymin[®] SK addition on the retention of AKD dispersion in pulp containing GCC (dispersion "A"; 1,25mg AKD/g fibre)

The negative interference between the strongly anionic filler and the cationic retention aid can be explained by the high affinity between both components. Due to the adsorption of polymer on the filler surface, the amount of polymer which is available for additional retention of AKD by coflocculation of fines, fibres and AKD particles is reduced.

Sequence of addition

High affinity of AKD particles to filler particles is responsible for the degradation of AKD [16; 17]. As a result spreading of AKD takes place on the surface of filler particles and ketone is formed as the final product of hydrolysis and decarboxylation reactions (Figure 10).

This time-dependent degradation of AKD is an industry-wide problem known as fugitive sizing or size reversion, especially in the case of PCC [15]. With PCC the residual amount of $Ca(OH)_2$ is believed to play an important role for ketone formation.

Therefore is was interesting to study the behaviour of GCC and PCC in a four component system in relation to the amount of ketone developed after sheet forming and drying. Thereby the ketone content stands as an indirect parameter for the adsorption of AKD (after the spreading process) on the filler particle. Regarding the results obtained above in the four component system the sequence of addition should exhibit a difference in the total ketone content.



Figure 10: Ketone formation from interaction filler/AKD

Experiments were performed in a four component system containing bleached sulfite pulp (8g/l), dispersion "A" (0,2 wt.%), anionically modified GCC or rod-shaped PCC (12,5 wt.%) and two different cationic polyelectrolytes: Solvitose[®] 2N and Polymin[®] SK.

Adding PCC as a filler to the fibre suspension, then dispersion "A" and finally the cationic polyelectrolyte, the highest degree of degradation and ketone formation was found. Compared to GCC the amount of ketone was 2-3 times higher (Figure 11).

Sequence of Addition

fiber / filler / dispersion "A" / retention aid



Figure 11: Comparison GCC vs. PCC, "wrong order of addition"

Adding first dispersion "A" to the fibre suspension, then the cationic polyelectrolyte and finally the filler, the extractable amount of ketone in both cases, using GCC or PCC, is in the same range (Figure 12). This experimental result clearly shows the extreme importance in the order of addition of wet end chemicals and the complex interactions between the charged species.

Sequence of Addition

fiber / dispersion "A" / retention aid / filler



Figure 12: Comparison GCC vs. PCC, "right order of addition"

We propose from these results that the adsorption of AKD particles to the filler surface or the exposure of AKD molecules to reactive sites on the filler surface can be reduced by preadsorption of cationic polymers like polyethylenimine or cationic starch onto the filler surface.

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AKD retention under practical conditions

As sizing agents dispersions "A" and "B" were used in the concentration range between 0,05 wt.% to 0,42 wt.% AKD. A good correlation was found between retention and the degree of sizing. In the case of system (1), the combination of TMP and CP, both dispersions show an almost quantitative retention giving reason for a good sizing level at addition of about 0,12 wt.% AKD (Figure 13).



Figure 13: Sizing level vs. AKD addition in system (1)

With system (2), the recycled paper as fibre source, the highly cationic dispersion "B" shows again an excellent retention and sizing, whereas the starch-stabilised dispersion exhibits a significantly lower retention (Figure 14). Therefore sizing develops much slower; even when twice the amount of AKD is added, the sizing level of dispersion "B" could not be reached.



Figure 14: Sizing level vs. AKD addition in system (2).

Conclusions

A new, reliable technique for the analysis of AKD in aqueous medium has been developed at BASF with the aim to determine the non-retained amount of AKD on cellulose fibres. Using this new technique we were able to study the interaction between fibres, fillers, the AKD dispersion and other process chemicals and their influence on the retention of AKD particles.

In a two component system containing only fibre and AKD dispersion the retention of AKD correlates very well with the electrophoretic mobility of the dispersion particles. A mobility of about 2 (μ m/s)/(V/cm) is sufficient for quantitative retention. The low retention of starch-based AKD dispersions can be greatly enhanced by addition of retention aids.

AKD dispersions show a high affinity to fillers like GCC and PCC therefore leading to a significant increase of AKD retention in a three component system as long as the filler retention is high.

The additional presence of retention aids in a three component system does not lead to the expected enhancement of AKD retention due to the consumption of retention aid by adsorption onto the filler surface.

Adsorption of AKD on filler surfaces leads to the formation of ketone. In order to minimise the formation of ketone, preadsorption of cationic polymers onto the fillers is proposed.

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Transcription of Discussion

Physico-chemical aspects of AKD retention and sizing efficiency

Anton Esser, BASF, Germany

Professor Tom Lindström, Royal Institute of Technology, Sweden

Can you comment on the different types of experimental technologies you are using. For instance, how is the emission or quantum efficiency affected by the state of agglomeration, the amount of fillers etc. It appears you are not willing to reveal the experimental procedures. Secondly, in your presentation you added the dye to the AKD before you added it to the stock and in your text you added the dye to the filtrate so could you illuminate the experimental procedures?

Anton Esser

In the textbook we described a procedure where we used a dynamic drainage jar for filtration. We found it was very hard to compare the conditions in the DDJ to the conditions of a handsheet maker. So it would be difficult to compare the results achieved with the DDJ to the results achieved with the handsheet maker. So we thought we had to have a method where we can measure directly the retention from the white water of the handsheet machine. Under these conditions the AKD is diluted to a much higher degree. We had to increase the sensitivity of our method and we found that the required sensitivity cannot be achieved by a method where you add the dye afterwards because you always have wood extractives in the pulp, which are also hydrophobic, and therefore they can be also dyed and give rise to a background fluorescence signal. We thought about what we could do to avoid the dyeing of other substances in the pulp. My colleague Dr Ettl put the dye directly into the wax before the formulation of the AKD emulsion. Using this technique you can increase the sensitivity towards AKD a lot. Then we can do the measurements on the handsheet maker. This is the reason why I described this improved method here in my presentation.

Professor Robert Pelton, Senior Scientist, McMaster University, Canada

Your last example involved TMP pulp. We all know that lignin is a strong fluorescing entity. Is there not a problem of interference with lignin in the TMP fines in your method?

Anton Esser

There is always a background fluorescence and you have to put enough dye into these particles so that the signal you get is higher than the background which is even fluctuating. That's also the reason why we can neglect the effect of fillers when we dilute the system down to a certain level, then the effect of fillers is smaller than the fluctuation of going from one measurement to another. This is the reason why we thought we could be independent of the effect of fillers from a practical point of view.

Ilkka Kartovaara, R&D Vice President, Enso Group, Finland

Many years ago we did some work which suggested that it is not only the level of retention but it may also be the nature of retention which influences the results so for example if you use a retention system which gives you more agglomeration of the size you might have poorer results. My question is did you try any retention aids other than polymene SK or did you find any indication about whether the nature of the retention would influence the results?

Anton Esser

First of all yes, we have used different retention aids. We used cationic starch and I think this is also described in the book. We also used polyacrylamide but I didn't present those results here because it would have been too much. One thing which was very interesting to us was the situation, when you compare two different modes of AKD addition. If we compare the situation where we add a lot of AKD without retention aid, with the situation where we add a normal amount of AKD together with retention aid, we find the same sizing efficiency for both situations. Although the mechanisms of adsorption are different we find that efficiencies are the same.

Nils-Olaf Bergh, Senior Adviser, Cerestar, Germany

Mr Kartovaara said, that he believes there is a question in which way AKD is agglomerated in the sheet and certainly he is correct in this. This is why I ask my question. In which way did you dry your sheets? Because, if you dry your sheet at high temperatures you always find too good sizing values (compared to paper machine produced paper) when the AKD is present in the sheet as bigger agglomerates. However, if you use retention aids, which are not so strongly flocculating you can achieve smaller particles of AKD, well distributed throughout the sheet. The sizing is in the latter case better. You cannot find differences in size retention or sizing level of interest to the papermakers if you use high temperature drying in the laboratory. The Cobb-method is not a good method to differentiate the effect of sizing agents at higher sizing levels.

Anton Esser

Yes I will accept this as an interesting subject for further research. I agree, we have a very high temperature when we cure our sheets.