The background features two thick, light green diagonal stripes that intersect to form a large 'V' shape. One stripe runs from the top right towards the bottom left, and the other runs from the top left towards the bottom right.

A note on AKD-sizing

An investigation of real and apparent contradictions in literature regarding spreading/diffusion of AKD on cellulose

Tom Lindström AND Tomas Larsson

December 2005

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Tom Lindström and Tomas Larsson

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1 Summary

Recent articles on Alkyl Ketene Dimer (AKD) sizing have challenged the classical view that sizing occurs in three consecutive steps, namely: size retention, size migration and finally a chemical reaction with cellulosic surfaces. Among other things it has been suggested AKD does not spread on cellulose and does not react with cellulose. Various alternative mechanisms such as vapour sizing and capillary wicking have also been suggested.

In this investigation direct experimental evidence for spreading/diffusion of AKD between couched sheets is given. It is concluded that spreading is a true macroscopic phenomenon and not only a microscopic event. It is also shown that no AKD can migrate in the vapour phase at $\text{temp} \leq 110^\circ \text{C}$.

Finally, it is suggested that some contradictory data in literature are only apparent contradictions. Hence, the lack of complete wetting of cellulose with AKD can be explained by published data suggesting an autophobic spreading sequence of AKD on cellulose combined with the way contact angles have been determined experimentally. The communication argues that many observations can be reconciled within the classical theoretical framework of AKD-sizing and that it is not necessary to invoke new mechanisms and that the classical sizing mechanism has the best power to interpret current scientific data available on AKD-sizing.

Sammanfattning

Nyligen publicerade artiklar rörande hydrofobering med Alkyl Keten Dimerer (AKD) har utmanat den klassiska bilden av att hydrofoberingen sker i tre konsekutiva steg, nämligen retention av hydrofoberingsmedlet, migrering (spridning) av hydrofoberingsmedlet samt slutligen en kemisk reaktion med cellulosa. Bland annat har det hävdats att AKD inte sprider sig på cellulosaytor och att AKD inte reagerar med cellulosan. Olika alternativa mekanismer såsom gasfashydrofobering, kapillär-inducerad spridning har också föreslagits.

I denna undersökning ges direkt experimentell bevisning för att spridning av AKD kan ske mellan pressade och hogguskade pappersark. Detta innebär att AKD-spridning är ett makroskopiskt fenomen och inte enbart en lokal mikroskopisk händelse. Det visas också att ingen AKD migrerar via gasformen vid temperaturer $\leq 110^\circ \text{C}$. Slutligen föreslås det att en rad motsägande data enbart är av chimär natur.

Således kan man förklara avsaknaden av total vätning av cellulosa med en i litteraturen föreslagen autofob spridningsmekanism kombinerat med det experimentella utförandet av kontaktvinkelmätningar.

Artikeln argumenterar för att de flesta experimentella observationer är fullt förenliga med klassik teoribildning för AKD-hydrofobering samt att det inte är nödvändigt med nya synsätt på hydrofoberingsförloppet samt att den klassiska teorin har bäst förmåga att på ett samlat sätt förklara tillgängliga vetenskapliga data rörande AKD-hydrofobering.

2 Introduction

AKD is the most important synthetic sizing agent used in the world paper/board industry to impart strong water repellence of paper. In view of its technical importance, the mechanism of sizing with these emulsions/dispersions has been the subject of numerous studies, which have been fairly recently reviewed by several authors (e.g. Hodgson 1994; Reynolds 1989; Roberts 1997). All these reviews are consistent with the current view that sizing occurs in three steps, namely: size retention, size migration and finally reaction with cellulosic surfaces. This general mechanism was extensively investigated by studies in this laboratory during the 80's using ^{14}C - labelled AKD-dispersions. (Lindström et al 1986 a-d).

More recent investigations have, however, challenged the traditional view. Hence, Garnier et al (1998, 1999, 2000) have found that AKD only wets, but not spreads, on cellulose and claim vapour phase sizing as a sizing mechanism for AKD. A capillary wicking mechanism was also suggested by Garnier and Godbout (2000).

Shen et al have also in a number of papers (e.g. 2001a,b, 2002, 2003) claimed that the classical view has been proven wrong and advocate mechanisms along the lines of Garnier and co-workers. These authors also claim there is no reaction between cellulose and AKD. Later investigations from this group (Hutton and Shen 2004), however, showed vapour phase sizing to be an insignificant phenomenon in practical papermaking sizing. This will also be confirmed in this investigation.

It is not the objective of this contribution to argue against these authors as the critical scientist may interpret the results as they stand in cited publications. On the other hand it is important to discuss scientific inconsistencies, which need to be rectified by continued scientific endeavours.

It is also suggested that some of the results reported in literature can actually be understood and be reconciled if the autophobic spreading mechanism suggested by Seppänen and Tiberg (2000) is taken into account, although these authors considered spreading as the rate determining step instead of the AKD-cellulose reaction.

Finally, a series of experiments illustrating, the ease of AKD-spreading and reaction in stacks of sheets are being reported. It is argued that the classical scheme of sizing events does not need to be revised.

2.1 Materials

The pulp used in all experiments was a never-dried unbeaten ECF (Elementary Chlorine Free) -bleached softwood (spruce/pine) kraft pulp (M-real, Husum, Sweden). A Celleco-filter with 100 μm screening slots was used to remove the fines (20-25 %) prior to the experiments. Before use, the pulp was first transferred to its H-form using 0.01 M HCl, after which the pulp was transferred to its Na-form using 10^{-3} M NaHCO_3 for 10 minutes. The cationic polyacrylamide (C-PAM) used was labelled PL1520 and was obtained from EKA Chemicals (Bohus, Sweden). This

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polymer had a molecular weight of 7 million and a 20 % molar charge density of cationic groups (according to the manufacturer).

2.2 Materials manufacture and treatments

2.2.1 Preparation of ¹⁴C-labelled AKD-dispersion

Standard ¹⁴C-labelled alkyl ketene dimer (AKD) (mp: 58-62° C) was made from a mixture of AKD wax (EKA Chemicals, Sweden) and ¹⁴C-labelled AKD wax (Amersham Pharmacia Biotech, Buckinghamshire, England). The dispersion was stabilized with a mixture of cationic starch (C-starch), HiCat 21370 (Roquette Freres, France), and lignosulfonic acid (LISA), Wanin S (Lignotech, Borregaard, Norway), according to a standard procedure given by EKA Chemicals reported previously (Johansson and Lindström, 2004). The fatty acid distribution was C18: 65 %, C16: 30 % and C14: 5 %.

2.2.2 Sheet forming

Hand sheets were made according to the SCAN C 26:76 standard. The stock was mixed in a vaned Britt Dynamic Drainage Jar (BDDJ) at a speed of 750 rpm. The pulp consistency was 0.342 %. The AKD was added to the stock and after 60 s stirring, the stock was poured into a British sheet former maintaining a total volume of 8 l and a pulp consistency of 0.171 %. This procedure is identical as the procedure labelled "modified procedure" in a previous publication (Johansson and Lindström 2004b).

Sheets were formed from pulps in their Na-form.

Note that all experiments were carried out in the presence of 1 mM NaHCO₃ (pH = 8). The HCO₃⁻ was added as a catalyst for the AKD-cellulose reaction.

The grammage of the hand sheets were 60 g/m². The individual sheets were first pressed between blotting papers at 3.4 bars for 5.5. min. Then 3 sheets (two outer plies without AKD-one middle sheet containing AKD) were stacked and pressed again between blotters. In a separate serie of experiments a thin teflon wire was inserted between the sheets in order to limit spreading/diffusion but allow for possible vapour phase migration.

Two drying procedures were employed. Photodrier at 90° C/10 min or at room temperature (25° C) during 24 hrs. Sheets dried at room temperature were then cured at 90° C/10 min. All sheets were finally post-cured at 110° C/10 min.

3 Analytical Methods

3.1 Tetrahydrofuran extraction of handsheets

The determination of the reacted amount of AKD was made in the following way:

Every hand sheet was extracted with tetrahydrofuran (THF) with the purpose of removing unreacted AKD. The hand sheets were first soaked in deionized water for 1h, 100 ml/g sheet, and thereafter soaked in THF, 100 ml/g sheet, for 1 minute. The water treatment step is necessary in order to swell the fibres so THF has access to fibre-fibre bonds where residual AKD is present, as has been discussed earlier (Lindström and Söderberg 1986a).

The sheets were then extracted in THF using a soxhlet for 6 cycles, air-dried and then reconsolidated in deionized water.

3.2 AKD retention and reaction

The extent of AKD retention and reaction of each hand sheet was determined by measuring the radioactivity of the sheets before and after THF extraction. The activity was determined by first oxidizing a piece of a hand sheet in a Packard Sample Oxidizer Model 307 (Hewlett Packard, USA).

The $^{14}\text{CO}_2$ was absorbed in Carbosorb E (Packard BioScience Company, USA) and diluted with Permaflour E+ (Packard BioScience Company, USA) before the ^{14}C -activity was measured in a Tri-Carb Liquid Scintillation Analyzer Model 2100TR.

4 Results

A simple set of experiments was designed in order to evaluate spreading between paper sheets. First it should be noted that the ^{14}C -labelled carbon is the C18, (see fig. 1) on the AKD, which means that if the AKD is hydrolysed, the total radioactivity is decreased. This is actually a suitable method to determine extent of hydrolysis of AKD, a subject to be covered in future publications from this laboratory. Evaporation of AKD, would also lead to a decrease in radioactivity, but experiments, where AKD wax is applied to dry paper and subsequently heated, there is no decrease in the radioactivity at temperatures below 110°C . The issue of possible vapour phase migration is also covered below.

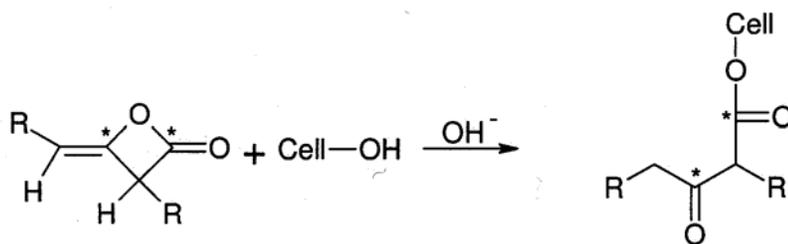


Figure 1 Reaction between AKD-cellulose ^{14}C -labelling position indicated (*).

Four sets of experiments were conducted in order to evaluate the transfer of AKD between stacked sheets.

First a control experiment (No 1 - see table 1), where AKD (1.5 mg/g) was added to the pulp (bleached softwood kraft pulp), after which a suitable retention aid (a cationic polyacrylamide, C-PAM, 0.05 %) was added to retain the AKD. All sheets were manufactured at $\text{pH} = 8.0$, with an added sizing catalyst (1 mM NaHCO_3). The AKD-retention was found to be 76.7 %, of which 40 % reacted with the cellulosic fibres after drying at 90°C .

In a second set of experiments (No 2.1-2.3), a sized sheet (as in exp. 1) was wet-pressed and placed between two unsized sheets (which had also been wet-pressed) as outer plies. The stack of three sheets was subsequently couched together in a second wet-pressing operation. This stack was then dried at room temperature for 24 hrs, after which the stack was cured at 90°C for 10 min.

A third series of experiments (3.1-3.3) were conducted where the stack of sheets were dried directly at 90°C . The dried stack of sheets could then be separated and the total AKD content could be determined, together with the reacted amount of AKD.

Finally, one serie of experiments was conducted, where a thin teflon wire was inserted between the sheets before couching in order to prevent size migration, but allow for possible vapour phase transfer of AKD.

It has already in early communications (Lindström and Söderberg 1986a) been concluded that under these laboratory conditions no hydrolysis of AKD takes place, when AKD-sized sheets were dried at temperatures above 90° C.

The retained amount of AKD in the sheets dried at 25° C, were determined by drying sheets directly at 90° C.

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Table 1 Results from AKD-transfer experiments. AKD-sized sheets (2.2 and 3.2) were stacked between unsized sheets (2.1/2.3 and 3.1/3.3) after wet pressing and subsequently dried at 25° C (serie no 2) or 90° C (serie no 3). After drying the sheets were post cured at 110° C for 10 min. One serie of experiments were conducted as 3.1-3.3 but a thin teflon wire was inserted between the sheets before couching to prevent size migration but allow for possible gas phase transfer of AKD.

Sheet Exp.no/ Sheet type	Content	Drying	AKD in Sample (mg/g)	AKD Retention (%)	AKD Reacted	
					(mg/g)	Reacted amount (%)
1.1 (Reference)	AKD (1.5 mg/g) + C-PAM	90° C	1.15	76.7	0.46	40.0
2.1	no AKD	25° C	0.16 ^a	75.3	0.09	56.3
2.2	AKD (1.5 mg/g) + C-PAM	"	0.79 ^a		0.23	29.1
2.3	no AKD	"	0.18 ^a		0.08	44.4
3.1	no AKD	90° C	0.16		0.07	43.8
3.2	AKD (1.5 mg/g) + C-PAM	"	0.88	80.0	0.38	43.2
3.3	no AKD	"	0.16		0.06	37.5
4.1	no AKD	90° C	<0.0005	-	-	-
4.2	AKD (1.5 mg/g) + C-PAM	"	1.06	70.0	n.d	n.d
4.3	no AKD	"	<0.0005	-	-	-

a) Determined by drying at 90° C - see text.

The following conclusions can be drawn from table 1.

Firstly, the total amount of AKD is, as expected constant, if adding the individual contributions from the different layers sums up the retained amount of AKD. This is expected, if there is no gas-phase transfer of AKD. The results show that AKD spreads readily to the outer plies when dried at 90° C (wet or dry treatments), although there is a lower amount of AKD in the outer plies. The amount of reacted AKD in the different plies is approximately constant, around 40 %, for the sheets dried at 90° C. For the sheets dried at 25° C, the reacted amount of AKD is somewhat lower in the middle-layer, which may be attributed to some hydrolysis of AKD, when stored at 25° C for 24 hrs before drying. The outer plies have a higher reacted amount, which may indicate higher spreading coefficient for AKD than for the hydrolysis product, the ketone.

Finally, from the experiments 4.1-4.3 it can be concluded that no vapour phase transport between the different sheets take place.

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5 Discussion

This contribution shows beyond any doubt that spreading of AKD is not only a microscopic phenomena but occurs in the macroscale allowing AKD-transfer even between different plies in a multilayer sheet construction. Moreover, there are also direct AFM-observations of AKD-spreading under various temperature conditions available in literature (Horn 2001). A central issue is how these facts can be reconciled with the observations by Garnier and co-workers (1998, 1999, 2000) that AKD spreading does not take place and that AKD forms a non-zero contact angle with cellulose?

Firstly, the thermodynamic conditions for spreading on cellulose may be considered.

The free energy of spreading for AKD on a cellulose surface can be written:

$$\Delta G_s = A(\gamma_{\text{Cell/AKD}} + \gamma_{\text{AKD}} - \gamma_{\text{Cell}}) \quad (1)$$

Where γ_{AKD} and γ_{Cell} is the surface free energy of AKD and cellulose respectively and $\gamma_{\text{Cell/AKD}}$ is the interfacial free energy between cellulose and AKD and A is the surface area.

Different authors have determined different values for the surface energy of cellulose, but consistent values have been obtained by inverse gas chromatography and a value for the total surface energy given by Lundqvist and Ödberg (1997) is 57.3 mN/m

The surface free energy of AKD is 27 mN/m at 75° C (Garnier and Godbout 2000) and using an equation of state for interfacial tensions by Neumann (1974):

$$\gamma_{\text{cell/AKD}} = \frac{|\sqrt{\gamma_{\text{AKD}}} - \sqrt{\gamma_{\text{cell}}}|}{1 - 0.015\sqrt{\gamma_{\text{AKD}} \cdot \gamma_{\text{cell}}}} \quad (2)$$

A value of $\gamma_{\text{Cell/AKD}}$ can be calculated to 5.7 mN/m.

This equation was derived for low energy surfaces and may not be directly applicable to cellulose/AKD interfaces, but $\gamma_{\text{Cell/AKD}} + \gamma_{\text{AKD}} - \gamma_{\text{Cell}}$ can now be estimated to -24.6 Nm/m. Hence, spreading should be expected.

The term “spreading”, if understood as a hydrodynamic phenomenon (in the context of the work by Garnier et al 1998) may not be the relevant term to describe AKD-migration on cellulose surfaces. Seppänen and Tiberg (2000) have, however, suggested that AKD-spreading takes place through an autophobic migration mechanism, which actually may resolve some of the apparent contradictions discussed in this paper.

Garnier et al (1998) came to the conclusion that AKD forms a decreasing contact angle with time, but still concluded the contact angle was non-zero, i.e. they

suggested AKD could not spread on cellulose. These authors assumed spreading was a purely hydrodynamic phenomenon and therefore these authors suggested that migration might take place by a capillary wicking mechanism. A capillary wicking mechanism was also discussed in later work by the same authors (Garnier and Godbout 2000). Although capillary wicking may be an active mechanism for AKD migration in paper structures, it cannot explain that very high amounts of reacted AKD under favourable conditions (68 %, Lindström et al 1986a), clearly suggesting migration involving AKD monolayers.

Later, however, Seppänen et al (2000) suggested AKD spreading occurred by a surface diffusion mechanism involving an autophobic monolayer precursor, which grows at the foot of the AKD/drop particle. Hence, if the contact angle of AKD on cellulose is non-zero, as was found in the work by Garnier et al (1988, 2000), size migration may take place from an AKD emulsion droplet through a surface diffusion mechanism as suggested by Seppänen et al (2000). These authors also concluded that the surface diffusion is a slow process, with an apparent diffusion coefficient of $1.1 \times 10^{-11} \text{ m}^2/\text{s}$ at 45° C . The authors, however, drew the wrong conclusion that the apparent surface diffusion was reflected in the time evolution of the development of hydrophobicity of paper during sizing, although the authors probably were perfectly right that migration of AKD is determined by surface diffusion of an autophobic monolayer. Indeed, a diffusion coefficient of the order of $10^{-11} \text{ m}^2/\text{s}$ was later confirmed by Shchukarev et al (2003), albeit at 80° C . Therefore, the time scale of the AKD-migration may now be estimated by using the apparent diffusion coefficient of $1.1 \times 10^{-11} \text{ m}^2/\text{s}$.

AKD-sizing particles typically have the dimension of the order of a micron. Assuming a radius of 0.5 microns, such a droplet would on a cellulose surface have a spreading time around 7 s, using this diffusion coefficient (Starting with a hemispherical drop of radius $0.5 \mu\text{m}$ and ending with a circular slice of mono-molecular thickness (10 \AA), both of equal volume, give a circular slice radius of about $9 \mu\text{m}$. (The time dependence in the drop radius is approximated by $r(t) = r_0 + \sqrt{Dt}$, which gives a time of about 7 s for the described increase in AKD drop radius). The time of reaction is in the order of 5 minutes (Lindström et al 1986a), hence spreading is not the rate-determining step in the consecutive events of sizing. It is therefore not necessary to invoke capillary wicking or size evaporation to explain AKD-migration. Akpabio and Roberts (1987) have also shown that vapour phase deposition does not lead to sizing because AKD is being hydrolyzed under such conditions. If autophobic surface diffusion in a monolayer is the governing spreading mechanism, this may also explain the non-zero wetting angles found by Garnier and co-workers. Under such circumstances, it will be essential to have a sufficiently large spreading area available, when determining the equilibrium contact angle between cellulose and AKD, so all the liquid in the drop can spread to a monomolecular layer if the contact angle actually is zero degrees. Otherwise migration will be constrained by the area available for surface diffusion. In the experiments conducted by Garnier et al (1998), the droplets used

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in the wetting experiments were $6 \mu\text{l}$ ($6 \times 10^{21} \text{ \AA}^3$). Assuming a thickness of a monomolecular film of AKD to be 10 \AA , a surface area of 6 m^2 would be required to form a monomolecular layer. This was certainly not the case in the experiments conducted by Garnier et al and that could explain why these authors only recorded finite contact angles. Hence, the apparent controversy may be resolved if the migration rate is determined by an autophobic monolayer.

The issue of reaction between cellulose and AKD and cellulose has also drawn some recent attention from Shen and co-workers (2001, 2003). In a fairly recent publication (Shen 2003) the authors claim that “the widely believed esterification reaction between AKD molecules and cellulose does not occur”, referring to four publications, invoking some kind of physical adhesion mechanism of an unknown nature. The authors are *not* citing the publications (^{13}C NMR: Bottorf et al 1993, 1994; ^{14}C -labelling/extraction: Lindström et al 1986a; FTIR: Ödberg et al 1987) usually considered as critical evidence for AKD-cellulose reaction in the review literature cited above. In our previous publications (Lindström et al 1986 a-b) the reaction (and kinetics of the reaction) between AKD and cellulose was studied using ^{14}C -labelled AKD. The effects of time, temperature, pH and various catalysts such as NaHCO_3 and polyamide amine resins were investigated. All these effects could be explained in terms of classical kinetic concepts. Moreover, the events were characterised with activation energies typical of chemical reactions. The reacted amount of AKD was determined by extracting non-reacted AKD and possible hydrolysis products using a procedure involving water-swelling, followed by extraction with tetrahydrofuran (THF). The effects of the catalysts are also readily understood from the chemistry and it would be very difficult to interpret catalysis in terms of effects on surface diffusion or some unknown type of adhesion mechanism. It was also shown that it is the reacted amount of AKD, which determines the sizing and that the specific surface area of the cellulosic fibres etc directly determines the sizing threshold. This is just to mention, that there is an overwhelming mass of observations in literature, which are readily understood in terms of classic concepts. These diverse observations must be reconciled by a theory, preferably understood in terms of classic physical/colloid chemistry. This is exactly what the classical concept referred to above (consecutive events of deposition/migration/reaction) does.

For these reasons, it is believed, that it is possible to explain all experimental findings regarding the action of AKD as a sizing agent within the “classical” previously published theoretical framework and there is no imminent need for an alternative theory.

Moreover, we have also shown that the cited apparent contradictions regarding AKD-migration possibly can be resolved if migration is limited by the surface diffusion of an autophobic layer.

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