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54 **PAPER SIZING COMPOSITIONS.**

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Description

The present invention relates to paper sizing compositions.

5 The use of sizing compositions for imparting a degree of water resistance to paper is well known. One such sizing composition comprises an aqueous dispersion of an alkyl ketene dimer (AKD) which has particular use in the preparation of paper containing chalk as a filler. This is because the AKD sizes are capable of effecting sizing under alkaline conditions in contrast to the generally acid condition (usually pH = 4 - 5.5) required by rosin emulsions.

10 AKD sizes do however have the disadvantage of being comparatively expensive to use due to the cost of the AKD itself.

It is an object of the present invention to obviate or mitigate the abovementioned disadvantage.

According to the present invention there is provided a paper sizing composition which comprises an aqueous dispersion of an alkyl ketene dimer characterized in that the dispersion incorporates urea, thiourea or derivatives thereof.

15 The invention further provides a method of preparing a sized paper comprising treating the paper or the fibrous stock (eg. cellulose fibres) from which it is prepared with a dispersion of an alkyl ketene dimer and with urea, thiourea or derivatives thereof. Preferably this method is effected by means of dispersion of the AKD which also includes the urea, thiourea (or derivatives). It is however within the scope of the invention to introduce the AKD dispersion and the urea (or thiourea or derivatives) as two separate components into the sizing process.

20 The invention also provides paper which has been sized with a composition in accordance with the invention.

The derivatives of urea and thiourea which may be used in accordance with the invention are those having a molecular weight up to 1000, and include salts of urea and thiourea.

25 The sizing compositions in accordance with the invention may be produced easily by admixing a dispersion of AKD (the production of which is well established technology) with a solution of urea (or thiourea or derivative). The resultant product may comprise 1 to 30 parts by weight (more preferably 1 to 18 pbw, most preferably 1 to 12 pbw, eg. 4 to 8 pbw) of alkyl ketene dimer per 100 pbw of dispersion together with an amount of urea (or thiourea or derivative) which is up to 2 times by weight that of the AKD. More typically, the dispersion will comprise an amount of urea, thiourea or derivative thereof which is from 5 to 20% by weight (more preferably 5 to 15%, most preferably about 10% by weight of the total weight of the AKD and urea, thiourea or derivative thereof).

Urea is the preferred compound for use in the invention because of its cheapness.

35 The compositions have been found to be storage stable and microscopic examination of the composition (after storage) has revealed no evidence of flocculation or separations.

The sizing compositions of the invention may also contain additional components as used in standard AKD emulsions. Such additional components include cationic starch, which may be used in an amount of 1-3%. Further components are surface active agents, eg. sodium ligno sulphate.

40 The invention has been based on our discovery that urea, thiourea and derivatives thereof may be used in partial replacement of the AKD in the dispersion without detriment to sizing properties. Since urea, thiourea and their derivatives are generally considerably cheaper than AKD itself, this obviously means that there is a cost saving as between an AKD dispersion in which the AKD is partially replaced by urea, thiourea or a derivative thereof and one in which there is no such substitution without there being less in sizing performance. Most preferably it is urea which is used as the partial replacement of the AKD.

45 Furthermore, trials over a period of several months have shown that the amount of an AKD dispersion of a particular solids content (dry basis) required for sizing a tonne of a particular type of paper may be greater than that required for a similar dispersion but in which the AKD has been partially replaced by urea, assuming of course that the quality of sizing is the same in both cases. The reason for this effect is not clear but may be due to the urea acting as a promoter for the cure of the AKD sizes. Indeed, in some cases (see Tables 2 and 3 infra) the Cobb Values of the 'as produced' paper is significantly higher in the case where urea is incorporated in the size than in the case where there is no such substitution. This suggests that urea is promoting curing. However this effect is not found in all sizing operations but, where it is present, overcomes a disadvantage of AKD sizes, namely that the sizing properties take a comparatively long time to develop in the finished paper. Thus, these properties may only become fully developed after storage for three days in the reel. This slow development of sizing properties is particularly a problem in paper making processes which involves the use of a size press. The comparatively low sizing of the paper by the time it reaches the press results in excessive pick up of the starch solution at the press and it is then necessary for the paper to stay on the drying cylinder for a comparatively long time. Thus production speeds need to be lowered to cater for the longer drying

time.

The invention will be further described by the following non-limiting Examples.

5 **Example 1**

An aqueous AKD dispersion comprising 5.4% by weight of AKD and 0.6% by weight of dissolved urea together with about 1.5% by weight of starch and about 0.3% surfactant was used over a three month period for the sizing of high quality printing and writing paper.

10 The average amount of the dispersion used (expressed as a percentage) per tonne of paper produced over this period is shown in the middle column of Table 1 below. For the purposes of comparison, the right hand column of Table 1 shows the corresponding figures for the same three months of the previous year during which sizing was effected with a dispersion comprising 6% by weight of AKD, about 1.5% by weight of cationic starch, and 0.3% by weight of surfactant.

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Table 1

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Month	Amount of Size Used	
	Invention	Comparison
1	1.99	2.65
25 2	1.77	2.30
3	2.05	1.90

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The quality of sizing obtained by using the composition of the invention was as good as that obtained with the comparison composition. Furthermore it will be noted from Table 1 that, taken over the three month period, the average amount of sizing composition used was less for the composition of the invention than that of the comparison composition. This obviously enhances the cost saving achieved by the substitution of part of the AKD by urea.

35 **Example 2**

An AKD dispersion of the following composition was prepared by standard techniques.

- 6% AKD
- 1.5% Cationic Starch (ca 20% moisture content)
- 40 0.3% Surfactant
- Balance Water

90 parts of this dispersion were blended with 10 parts of a 7.5% solution of urea.

The resultant product was used for sizing in the production of 200 g/m² caseliner.

45 The Cobb values of the 'as produced' paper were measured by standard techniques and are shown in the first column of Table 2 below. Additionally, samples of the 'as produced' paper were heated for 10 minutes at 105°C and the Cobb values measured. The results are again included in the second column of Table 1. This latter test corresponds to that used in the production of paper using AKD size without a curing agent where the 10 minute heating is used to simulate the storage of the paper of the reel for 3 days and thus give an indication of the quality of the sizing. Finally, the Cobb values of the paper from the reel were re-measured after
50 several days and are shown in the third column of the Table.

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TABLE 2

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Cobb Values

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	As Produced	Cured for 10 mins	Stored for several days
Reel 1	24	28	(1)
Reel 2	23	28	22.8
Reel 3	27	30	24

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(1) not measured

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It will be seen that the 'as produced' paper has a Cobb value in the 'hard sized' range. The values improve slightly on storage (column 3). Although the 'as produced' values seem superior than the over cured values (column 2) this is not particularly significant and may be accounted for by the lack of moisture in the over cured samples.

By way of comparison, Table 3 gives a data for caseliner produced with the AKD size without added urea.

TABLE 3

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Cobb Values

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	As Produced	Cured for 10 mins	Stored for several days
Reel 4	102	32	30.7
Reel 5	63	30	30.4

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It can immediately be seen that paper produced without urea in the AKD size has poor sizing properties in the 'as produced' paper condition.

It must however be mentioned that hard sizing in the 'as produced' paper such as demonstrated in Table 2 above is not obtained for the sizing of all papers with the AKD/urea size.

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Claims

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1. A paper sizing composition comprising an aqueous dispersion of an alkyl ketene dimer characterised in that the dispersion incorporates urea, thiourea or a derivative thereof.
2. A sizing composition as claimed in claim 1 comprising 1 to 30% by weight of the alkyl ketene dimer of the dispersion.
3. A sizing composition as claimed in claim 2 comprising 1 to 12% by weight of the alkyl ketene dimer of the dispersion.

4. A sizing composition as claimed in claim 3 comprising 4 to 8% by weight of alkyl ketene dimer of the dispersion.
- 5 5. A sizing composition as claimed in claims 4 comprising 5 to 15% by weight of urea based on the total weight of the AKD and urea.
6. A sizing composition as claimed in any one of claims 1 to 4 wherein the amount of urea, thiourea or derivative thereof is up to 2 times the weight of that of the AKD.
- 10 7. A sizing composition as claimed in claim 6 wherein the amount of urea, thiourea or derivative thereof comprises 5 to 15% by weight of the total weight of the AKD and the urea, thiourea or derivative thereof.
8. A method of preparing a sized paper comprising treating the paper or the fibrous stock from which it is prepared with a aqueous dispersion of an alkyl ketene dimer and with urea, thiourea, or a derivative thereof.
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Patentansprüche

- 20 1. Papierleimungszusammensetzung, die eine wäßrige Dispersion eines Alkylketen-Dimers umfaßt, dadurch gekennzeichnet, daß die Dispersion Harnstoff, Thioharnstoff oder ein Derivat davon enthält.
2. Leimungszusammensetzung gemäß Anspruch 1, die 1 bis 30 Gewichtsprozent des Alkylketen-Dimers der Dispersion umfaßt.
- 25 3. Leimungszusammensetzung gemäß Anspruch 2, die 1 bis 12 Gewichtsprozent des Alkylketen-Dimers der Dispersion umfaßt.
4. Leimungszusammensetzung gemäß Anspruch 3, die 4 bis 8 Gewichtsprozent des Alkylketen-Dimers der Dispersion umfaßt.
- 30 5. Leimungszusammensetzung gemäß Anspruch 4, die, bezogen auf das Gesamtgewicht des AKD und des Harnstoffes, 5 bis 15 Gewichtsprozent Harnstoff umfaßt.
- 35 6. Leimungszusammensetzung gemäß einem der Ansprüche 1 bis 4, bei der die Menge an Harnstoff, Thioharnstoff bzw. des Derivates davon ein bis zu zweimal so großes Gewicht aufweist wie das des AKD.
7. Leimungszusammensetzung gemäß Anspruch 6, bei der die Menge an Harnstoff, Thioharnstoff bzw. des Derivates davon 5 bis 15 Gewichtsprozent des Gesamtgewichts des AKD und des Harnstoffes, des Thioharnstoffes bzw. des Derivates davon umfaßt.
- 40 8. Verfahren zur Herstellung eines geleimten Papiers, welches folgendes umfaßt: die Behandlung des Papiers bzw. des Faserstoffes, aus dem es hergestellt wird, mit einer wäßrigen Dispersion eines Alkylketen-Dimers und mit Harnstoff, Thioharnstoff oder einem Derivat davon.
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Revendications

- 50 1. Composition d'encollage de papier comprenant une dispersion aqueuse d'un dimère d'alkyl-cétène, caractérisée en ce que la dispersion incorpore de l'urée, de la thiourée ou un dérivé de celles-ci.
2. Composition d'encollage selon la revendication 1, comprenant 1 à 30 % en poids du dimère d'alkyl-cétène de la dispersion.
3. Composition d'encollage selon la revendication 2, comprenant 1 à 12% en poids du dimère d'alkyl-cétène de la dispersion.
- 55 4. Composition d'encollage selon la revendication 3, comprenant 4 à 8 % en poids de dimère d'alkyl-cétène de la dispersion.

5. Composition d'encollage selon la revendication 4, comprenant 5 à 15% en poids d'urée par rapport au poids total de dimère d'alkyl-cétène et d'urée.
- 5 6. Composition d'encollage selon l'une quelconque des revendications 1 à 4, dans laquelle la quantité d'urée, de thiourée ou de dérivé de celles-ci est jusqu'à 2 fois en poids celle du dimère d'alkyl-cétène.
7. Composition d'encollage selon la revendication 6, dans laquelle la quantité d'urée, de thiourée ou de dérivé de celles-ci comprend 5 à 15% en poids du poids total du dimère d'alkylcétène et de l'urée, de la thiourée ou de dérivé de celles-ci.
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8. Méthode de préparation d'un papier encollé comprenant le traitement du papier ou de la pâte à papier fibreuse à partir de laquelle il est préparé en utilisant une dispersion aqueuse d'un dimère d'alkyl-cétène et en utilisant de l'urée, de la thiourée, ou un dérivé de celles-ci.
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