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(54) Title: DISULFO-TYPE FLUORESCENT WHITENING AGENTS

(57) Abrégé/Abstract:
The present invention relates to the use of concentrated aqueous fluorescent whitening agent preparations for optically whitening paper, wherein the preparation contains a specific disulfo-type fluorescent whitening agent.

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(54) Title: DISULFO-TYPE FLUORESCENT WHITENING AGENTS

(57) Abstract: The present invention relates to the use of concentrated aqueous fluorescent whitening agent preparations for optically whitening paper, wherein the preparation contains a specific disulfo-type fluorescent whitening agent.

Disulfo-type fluorescent whitening agents

The present invention relates to the use of specific disulfo-type fluorescent whitening agents for whitening paper or board.

It is well known that the whiteness of paper and board can be improved by the addition of fluorescent whitening agents (FWAs). The most important FWAs used in the paper and board industry are anilino-substituted bistriazinyl derivatives of 4,4′-diaminostilbene-2,2′-disulfonic acid (flavonic acid). From these FWAs disulfo-, tetrasulfo- and hexasulfo-types are known. The disulfo-type FWAs with no sulfonic acid groups at the aniline rings have a low solubility in water and a high affinity for cellulose fibres. They are especially suitable for use at the wet-end of paper making process. The hexasulfo-type FWAs with two sulfonic acid groups at each aniline ring have a high solubility in water and a low affinity for cellulose fibres. They are more specialty products when very high whiteness is desired. The tetrasulfo-type FWAs with one sulfonic acid group at each aniline ring exhibit a behaviour between the disulfo- and hexasulfo-type FWAs and are most commonly used for whitening paper or board.

For ease of handling and metering, the paper and board industry demands FWAs to be supplied in a liquid form, preferably as a concentrated aqueous solution, which should be stable to prolonged storage over a wide temperature range. Due to the low solubility of disulfo-type FWAs in water, currently solubilising auxiliaries such as urea, triethanolamine or diethylene glycol are added in amounts of up to 30% to provide storage stability for concentrated aqueous solutions of disulfo-type FWAs. These solubilising agents have no affinity to cellulose and contami-

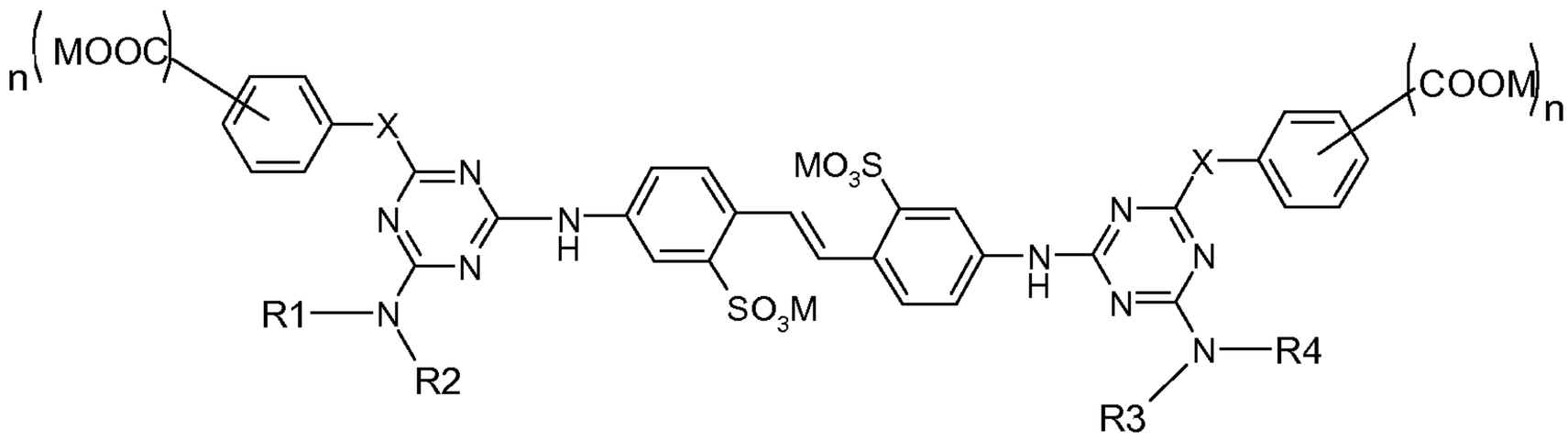
nate the effluent from the paper mill, thus being undesired. EP-A-1 752 453 teaches storage stable solutions of disulfo-type FWAs which contain specific counter-ions for the sulfonic acid groups, which counter-ions are derived from specific aminoalkanols. WO 02/055646 A1 discloses concentrated aqueous solutions containing a mixture of two specific disulfo-type FWAs.

Alternatively, slurries or dispersions of disulfo-type FWAs in water are known, e.g. from EP 0 884 312 B1. However, in order to enable the metering of homogenous preparations into the papermaking process, usually stirring is required.

Surprisingly, it has been found that problems of the prior art can be overcome by using concentrated aqueous preparations of specific disulfo-type fluorescent whitening agents having carboxylic acid groups at the aniline rings for whitening paper or board. These disulfo-type fluorescent whitening agents enable stable concentrated aqueous preparations or solutions to be formed, without addition of solubilising auxiliaries. Moreover, the production process of those fluorescent whitening agents is more cost-effective, compared to that of the commonly used disulfo-type fluorescent whitening agents, since it dispenses with laborious isolation and filtration steps.

Therefore, the present invention relates to the use of aqueous fluorescent whitening agent (FWA) preparations for optically whitening paper or board, wherein the fluorescent whitening agent preparation contains

(a) 5 to 80 % by weight of at least one fluorescent whitening agent (FWA) selected from the bis(triazinylamino) stilbene derivatives of the general formula (I)



(I)

wherein

X represents independently of each other O or NR´, where R´ is hydrogen or C_1-C_3 alkyl;

n is 1 or 2;

R_1, R_2, R_3 and R_4 represent, independently of each other, hydrogen, cyano, C_1 – C_4 alkyl, C_2 – C_4 hydroxyalkyl, or C_1 – C_4 alkoxyalkyl, wherein alkyl is linear or branched; or R_2 and R_1 or R_3 and R_4 independently of each other together with N atom form morpholine, piperidine or pyrrolidine ring; or $–(CH_2)_l$-SO_3M, where l is 1, 2 or 3; or $–(CH_2)_i$-COOR, $–(CH_2)_i$-CONHR, $–(CH_2)_i$-COR, where i is an integer from 1 to 4, R is C_1-C_3 alkyl or has the same meaning as M;

M represents hydrogen, or one equivalent of a cation, in particular Li, Na, K, Ca, Mg, ammonium, or ammonium which is mono-, di-, tri- or tetra-substituted by C_1 – C_4 alkyl or C_2 – C_4 hydroxyalkyl; and

(b) 95 to 20 % by weight of water.

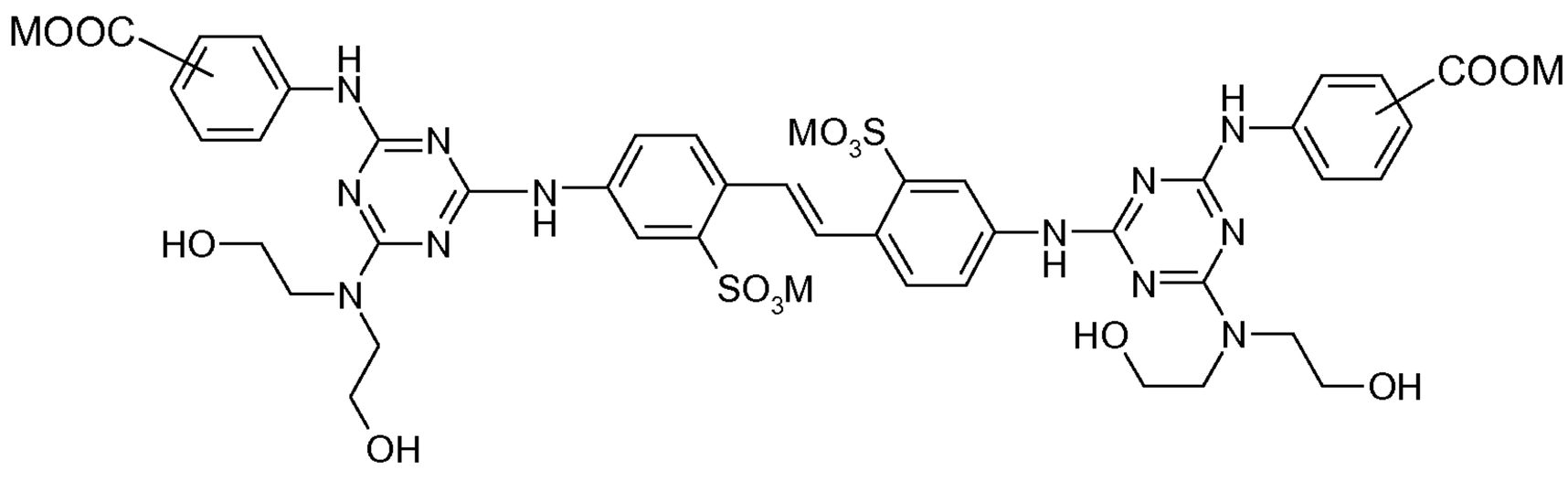
The invention also refers to the use of the aqueous fluorescent whitening agent (FWA) preparations for whitening paper in the pulp or at the surface. Further, the invention relates to a process for whitening paper and to paper obtainable by this process. Preferred embodiments of the invention are described in the description hereinafter, the figures and the claims.

Fig. 1 is a diagram showing the whitening performance of different fluorescent whitening agents in wood-free pulp.

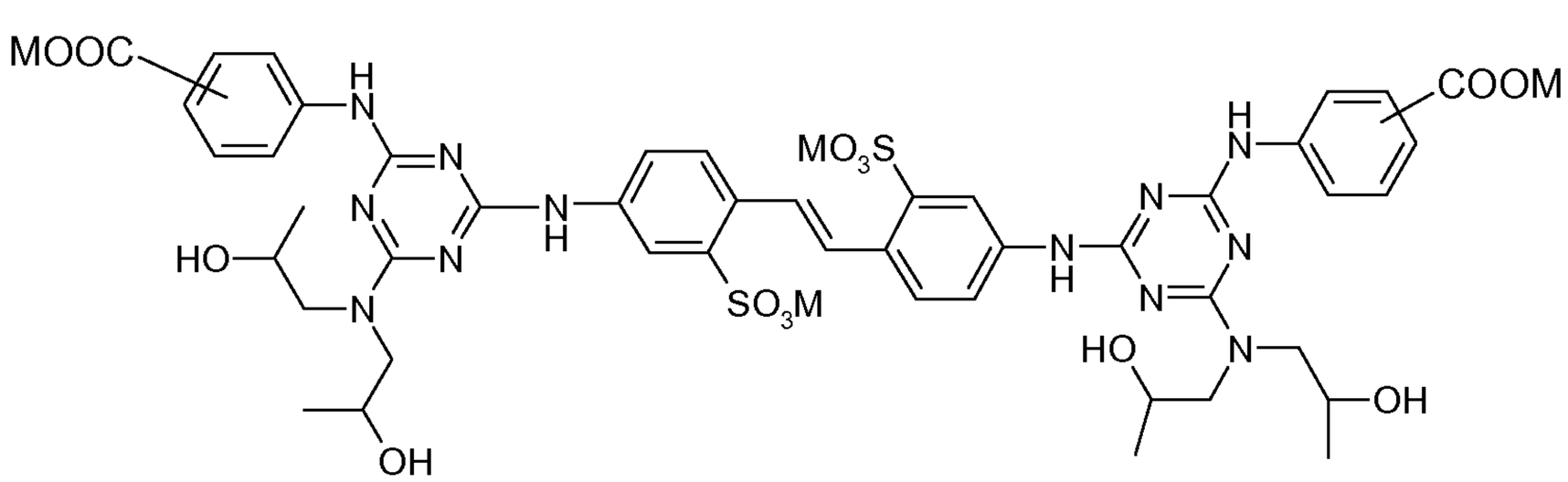
Fig. 2 is a diagram showing the whitening performance of different fluorescent whitening agents in wood-containing pulp.

According to the invention component (a) of the aqueous FWA preparation contains at least one FWA of the above defined formula (I). In a preferred embodiment, X represents NR´. In another preferred embodiment, n is 1. In a further preferred embodiment, R_1, R_2, R_3 and R_4 represent, independently of each other, C_2 – C_4 hydroxyalkyl, or C_1 – C_4 alkoxyalkyl, wherein alkyl is linear or branched Preferred embodiments of M are hydrogen, Na, K, Ca, Mg, in particular M is Na, K or hydrogen, most preferred is Na.

Preferred FWAs are the FWAs of following formula (Ia) and formula (Ib), wherein the carboxylic acid residues are, independently of each other, in *ortho*- or *para*–position, preferably in *para*–position:



(Ia)



(Ib)

The FWAs of formula (I) can be prepared by known procedures, and are used as free acids or as salts thereof, preferably alkali metal salts. Generally, the compounds are prepared by reacting cyanuric chloride with 4,4′-diaminostilbene-2,2′-disulfonic acid or a salt thereof, and an appropriate carboxyl acid group containing derivative, e.g. amino benzoic acid. PL patent 61710 discloses the preparation of some specific FWAs of the above defined formula (I) with one carboxylic acid group in p-position of each aniline ring. GDR (DDR) patent 55 668 discloses a further process for preparing some specific FWAs of the above defined formula (I) with one or two carboxylic acid groups at each aniline ring.

The purification of the FWAs of formula (I) is easier and thus more cost-effective than for commonly used disulfo-type FWAs, since isolation steps can be avoided. The purification could be carried out by, for example, membrane filtration. In contrast to the water evaporation or salt precipitation steps disclosed in PL patent 61710, the purification of the FWAs of formula (I) can be achieved by membrane filtration and the product obtained can be used as such. This is due to the surprisingly higher solubility of FWA of formula (I).

The aqueous FWA preparation used according to the invention can contain one or more FWAs of the formula (I). In a preferred embodiment, the preparation contains one FWA of the formula (I). In another preferred embodiment, the preparation contains two or three FWAs of the formula (I). It is also possible that other known FWAs are additionally used.

In a preferred embodiment, the aqueous FWA preparation used according to the invention contains 6 to 80 % by weight, preferably 7 to 80 % by weight, in particular 8 to 75 % by weight, more preferably 9 to 70 % by weight, most preferably 10 to 65 % by weight, of component (a). The water (component (b)) is preferably contained in an amount of 94 to 20 % by weight, preferably 93 to 20 % by weight, in particular 92 to 25 % by weight, more preferably 91 to 30 % by weight, most preferably 90 to 35 % by weight. In other preferred embodiments, the aqueous

FWA preparation contains 12 to 60 % by weight, preferably 15 to 55 % by weight, in particular 20 to 50 % by weight, of component (a), and 88 to 40 % by weight, preferably 85 to 45 % by weight, in particular 80 to 50 % by weight, of component (b). Unless otherwise indicated, the weight percentages herein are based on 100 % by weight of the aqueous FWA preparation.

In a preferred embodiment, the aqueous FWA preparations are free of crystalline whitener particles, in particular their hydrate forms.

The amount of component (a) in the aqueous FWA preparation may depend on the temperature of the preparation. Optionally, the aqueous FWA preparation used according to the invention may contain a small amount of auxiliaries. This might be particularly relevant for FWA preparations used in cold regions to enhance preparations' cold stability. In a preferred embodiment, the aqueous FWA preparation contains less than 25 % by weight, preferably less than 20 % by weight, more preferably less than 15 % by weight, in particular less than 10 % by weight of components other than components (a) and (b). For example, formulation auxiliaries, such as standardizing agents, surface-active compositions, antifoams, organic thickeners, preservatives, and /or electrolytes may be used. However, for ecological reasons, the aqueous FWA preparation preferably contains only very small amounts of components other than components (a) and (b), e.g. organic additives or auxiliaries, particularly altogether less than 3 % by weight, in particular less than 1 % by weight, based on 100 % by weight of aqueous FWA preparation. Particularly preferably, the FWA preparation contains no organic co-solvents, and/or urea. In a further preferred embodiment, the FWA preparation consists or consists essentially of components (a) and (b).

The aqueous FWA preparation is present in liquid form, in particular as a solution.

The aqueous FWA preparation is preferably prepared by introducing the at least one FWA of formula (I) in form of a powder or a concentrated solution thereof into water. Any auxiliaries can optionally be added during the preparation of the aqueous FWA preparation.

The aqueous FWA preparations can be used for whitening paper or board in the pulp suspension (stock) or pulp, in particular in the wet-end, or for applications to the surface. In wet-end applications the preparations can be added at any point of the pulp circuit, e.g. chests or pipes, before sheet forming. Depending on the papermaking process used, the preparations can be added to the papermaking process also in diluted form, wherein the preparation has been diluted to a desired concentration by addition of water and/or auxiliaries. In a preferred embodiment, the aqueous FWA preparation is introduced, optionally after dilution with water, to the pulp or pulp suspension. The preparations can be added continuously or discontinuously. The application is beneficial for both wood-containing pulps and wood-free pulps.

The aqueous FWA preparations exhibit high storage stability and ease of application. Simultaneously, they provide high affinity (substantivity) to fibres and high whitening performance.

The invention also refers to a process for whitening paper, which comprises providing a pulp or pulp suspension; adding the aqueous FWA preparation to the pulp or pulp suspension, preferably in an amount of 0.01 to 5 % by weight, more preferably 0.02 to 2 % by weight, based on dry pulp; producing a paper sheet from the pulp; and drying the sheet. The aqueous FWA preparation used in this process is the same aqueous FWA preparation as described above. In one embodiment of this process, the aqueous FWA preparation is added, after dilution with water and/or auxiliaries, in particular dilution with water, to the pulp or pulp suspension.

Paper produced by using the aqueous FWA preparations exhibits higher whiteness and higher greening limit compared to typically used disulfo-type FWAs.

The whiteness of the papers produced can be characterized by the CIE whiteness. Different FWAs can be compared to each other with respect to the saturation behaviour when determined according to CIE whiteness. In other words, if a larger amount of FWA is used and no further increase in whiteness is found, there is saturation behaviour and there may even be adverse effects on the whiteness when using higher amounts. The effect of saturation is also referred to as greening. The greening limit, i.e. the point at which increasing amounts of FWA used results in virtually no further increase in whiteness, can be derived, for example, from the a*-b* diagram, where a* and b* are the colour coordinates in the CIE-L*a*b* system.

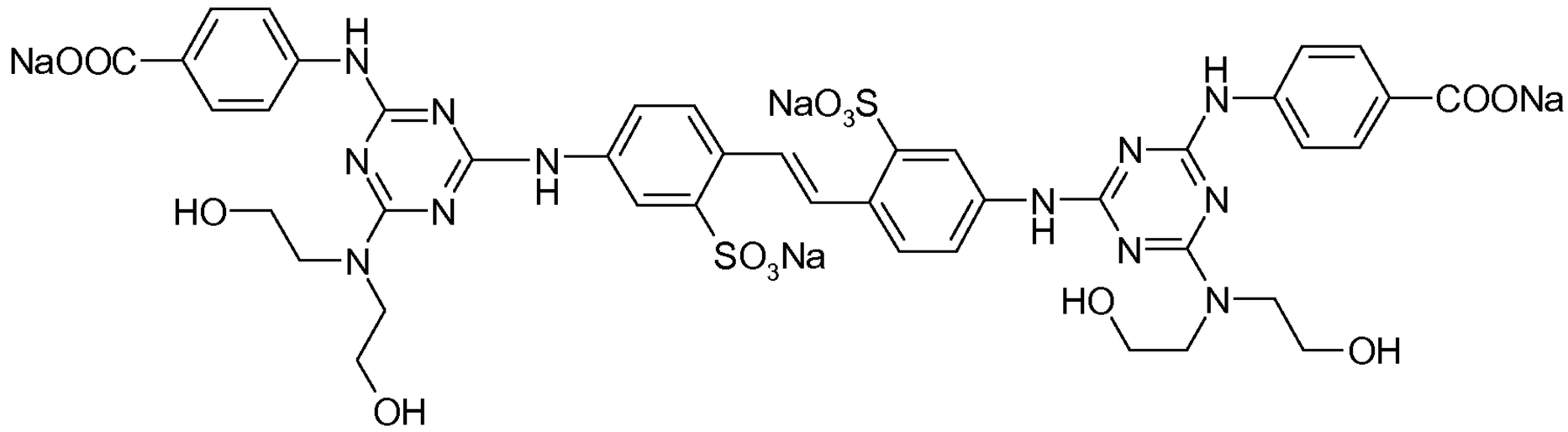
The following examples illustrate the invention and show preferred embodiments without limiting the scope of protection.

Examples

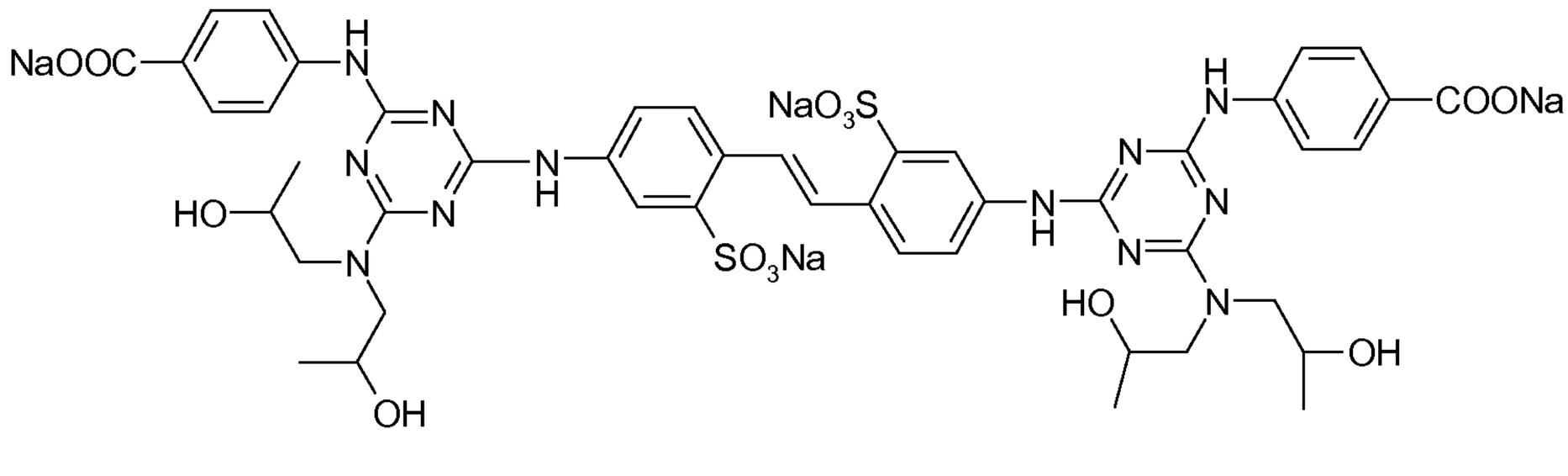
Example 1

The solubility behaviour of two fluorescent whitening agents used according to the invention and of a commonly used fluorescent whitening agent of the disulfo-type was studied. Further, the stability behavior of a concentrated aqueous solution was tested.

The tested fluorescent whitening agents of the invention were the following:

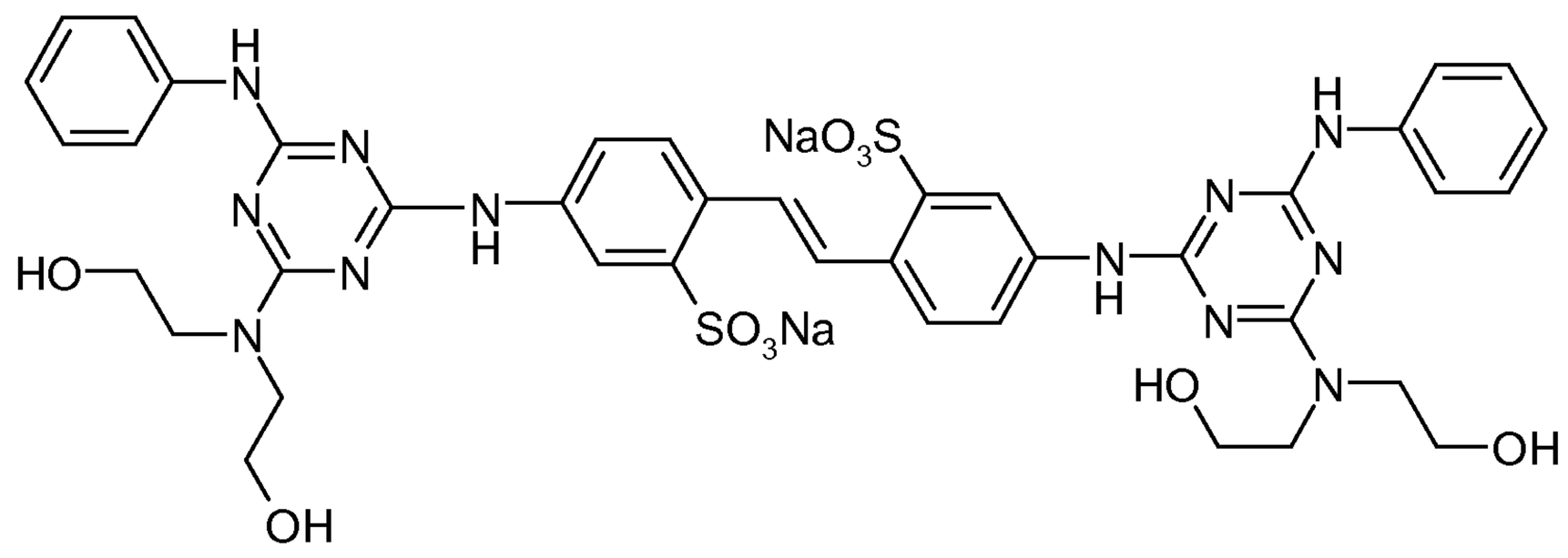


FWA 1



FWA 2

For comparison, the following, commonly used fluorescent whitening agent of the disulfo-type was used:



Comparative 1

The solubility of the fluorescent whitening agents was determined as follows: a dry powder of fluorescent whitening agent was added to 50 ml of distilled water until the saturation point was reached. The thus obtained saturated solution was filtered, dried and the dried residue was weighted.

The following results were obtained: 27.8 % for FWA 1; 24.2 % for FWA 2; and 6.9 % for Comparative 1, all at ambient temperature (about 22 °C). The indicated percentages are based on the amount of grams of fluorescent whitening agent dissolved in 100 g of the corresponding saturated FWA solution.

The stability behavior was studied by storing an approximately 20% solution of FWA 1 and FWA 2, respectively, at ambient temperature and at 4° C, each without stirring. The aqueous brightener preparations have a shelf-life of more than 30 days both at ambient and low temperature. They showed no crystalline precipitates.

Thus, FWA 1 and 2 exhibit a much higher solubility than Comparative 1. Simultaneously, concentrated solutions thereof exhibit high stability.

Example 2

The whitening performance of the FWAs of Example 1 was studied using the following test procedure.

The wood-free furnish (pulp suspension) was composed of 70 pts (parts, based on weight) of short fibres and 30 pts of long fibres with a grinding degree of 30-35°SR (Schopper-Riegler). The wood-containing furnish was composed of 50 pts of mechanical pulp, 35 pts of long fibres and 15 pts of short fibres with a grinding degree of 40 °SR.

800ml of a 0.625% of corresponding furnish were weighted in a beaker to prepare a 5g hand sheet of ~120g/m^2 for each experimental series. A 0.1 wt % FWA solu-

tion was prepared using distilled water. The amounts of FWA as indicated in Table 1 below were achieved by adding a corresponding amount of the 0.1 wt % FWA solution by a pipette to the stirred pulp suspension which was allowed to stir for 10 minutes after FWA addition. The amounts of FWA in Table 1 are calculated as active ingredient on 100 wt % of dry pulp.

A wet filter paper was positioned on the wire of the sheet former, the stock is put on the sheet former and sucked dry. The formed hand sheet was protected by an additional dry filter, pressed and dried on a calender at 100°C. Thereafter, the obtained hand sheets were equilibrated in a climate room under standard conditions overnight and then measured with a Datacolor spectrometer (ISO2469) by determining CIE, L*, a* and b*, the light source used based on ISO2469 standard.

The results obtained are summarized in Table 1 and further shown in the Figures. Fig. 1 shows the results for the wood-free pulp and Fig. 2 for the wood-containing pulp.

Table 1

FWA	Amount (wt %) FWA	CIE whiteness	L*	a*	b*
Wood-free pulp					
FWA 1	0.04	118.59	97.15	1.89	-5.83
	0.08	129.29	97.48	2.41	-8.10
	0.16	138.27	97.76	2.69	-10.03
	0.24	142.39	97.86	2.71	-10.92
	0.32	143.96	97.93	2.57	-11.25
FWA 2	0.04	117.27	97.22	1.86	-5.49
	0.08	127.23	97.46	2.36	-7.64
	0.16	136.69	97.68	2.74	-9.70
	0.24	141.11	97.80	2.87	-10.66
	0.32	143.91	97.74	2.86	-11.33

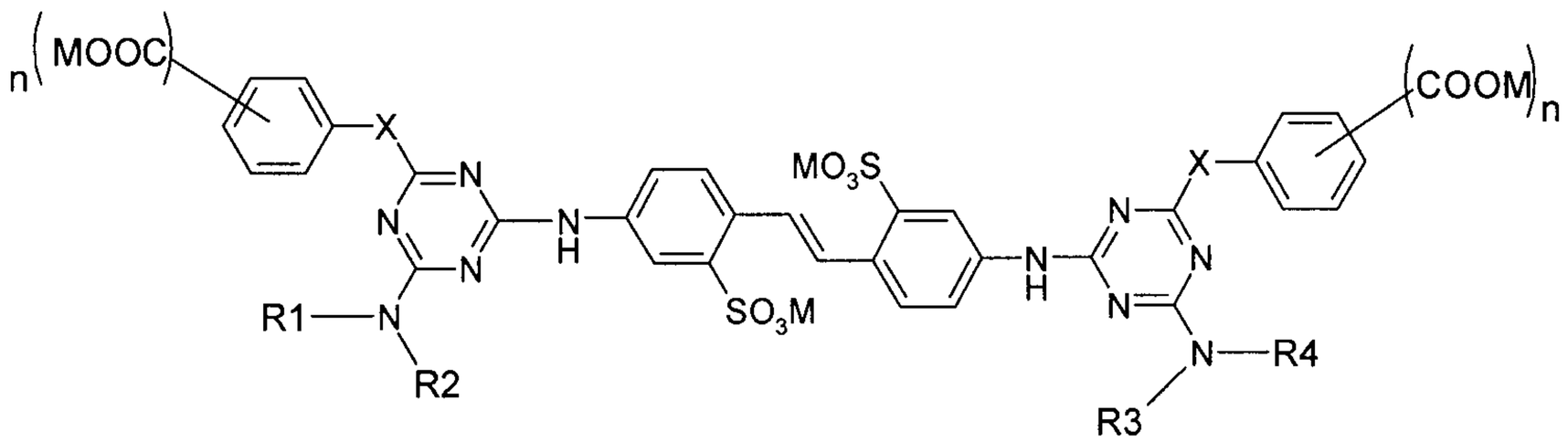
Comparative 1	0.04	115.09	97.13	1.71	-5.04
	0.08	124.22	97.30	2.18	-7.03
	0.16	134.02	97.61	2.54	-9.12
	0.24	138.27	97.81	2.58	-10.00
	0.32	140.10	97.89	2.51	-10.38
Wood-containing pulp					
FWA 1	0.04	52.12	93.83	-0.08	7.14
	0.08	59.30	94.08	0.22	5.72
	0.16	65.61	94.20	0.30	4.41
	0.24	68.43	94.31	0.22	3.85
	0.32	69.74	94.45	0.18	3.64
FWA 2	0.04	50.86	93.84	-0.10	7.42
	0.08	57.77	94.02	0.17	6.02
	0.16	64.23	94.09	0.33	4.65
	0.24	67.19	94.12	0.36	4.02
	0.32	68.76	94.31	0.35	3.78
Comparative 1	0.04	50.01	93.85	-0.11	7.61
	0.08	56.60	94.01	0.16	6.27
	0.16	62.58	94.18	0.26	5.06
	0.24	65.75	94.24	0.30	4.40
	0.32	67.23	94.32	0.29	4.12

Thus, the FWAs used according to the invention exhibit same or even better whitening performance than a commonly used disulfo-type FWA while simultaneously having higher solubility in water, thus allowing the preparation of stable, concentrated aqueous preparations for whitening paper or board.

CLAIMS

1. Use of an aqueous fluorescent whitening agent (FWA) preparation for optically whitening paper or board, wherein the aqueous FWA preparation contains

(a) 5 to 80 % by weight of at least one FWA, wherein the FWA is a bis(triazinylamino) stilbene derivative of formula (I)



(I)

wherein

each X represents independently of each other, O or NR′, where R′ is hydrogen or C_1-C_3 alkyl;

each n is, independently of each other, 1 or 2;

R_1, R_2, R_3 and R_4 represent, independently of each other, hydrogen, cyano, C_1 – C_4 alkyl, C_2 – C_4 hydroxyalkyl, or C_1 – C_4 alkoxyalkyl, wherein alkyl is linear or branched; or $-(CH_2)_l$-SO_3M, wherein l is 1, 2 or 3; or $-(CH_2)_i$-COOR, $-(CH_2)_i$-CONHR, or $-(CH_2)_i$-COR, where i is an integer from 1 to 4, R is C_1-C_3 alkyl or has the same meaning as M; or

R_2 and R_1 or R_3 and R_4 independently of each other together with an N atom form a morpholine, piperidine or pyrrolidine ring; and

each M represents independently of each other hydrogen, or one equivalent of a cation; and

(b) 95 to 20% by weight of water.

2. The use of the aqueous FWA preparation according to claim 1, characterized in that M represents Li, Na, K, Ca, Mg, ammonium, or ammonium which is mono-, di-, tri- or tetra-substituted by $C_1 - C_4$ alkyl or $C_2 - C_4$ hydroxyalkyl.

3. The use of the aqueous FWA preparation according to claim 1 or 2, characterized in that the preparation contains 10 to 65 % by weight of component (a).

4. The use of the aqueous FWA preparation according to any one of claims 1 to 3, characterized in that in the FWA, each X represents NR′.

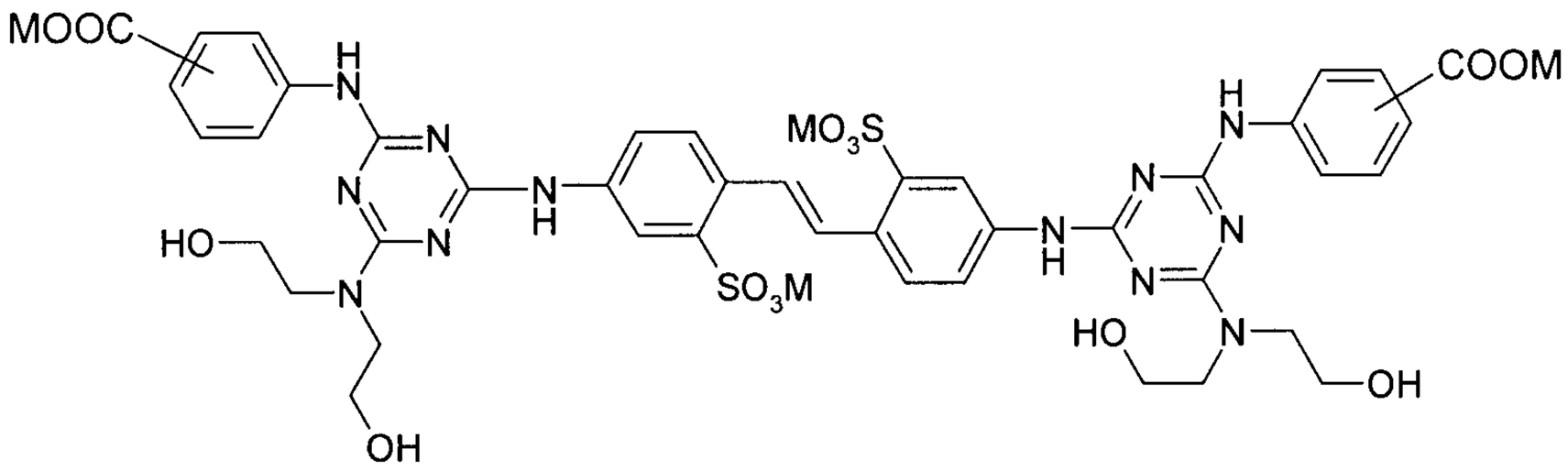
5. The use of the aqueous FWA preparation according to any one of claims 1 to 4, characterized in that in the FWA, n is 1.

6. The use of the aqueous FWA preparation according to any one of claims 1 to 5, characterized in that R_1, R_2, R_3 and R_4 represent, independently of each other, $C_2 - C_4$ hydroxyalkyl, or $C_1 - C_4$ alkoxyalkyl.

7. The use of the aqueous FWA preparation according to any one of claims 1 to 6, characterized in that the aqueous FWA preparation contains less than 10 % by weight of components other than components (a) and (b).

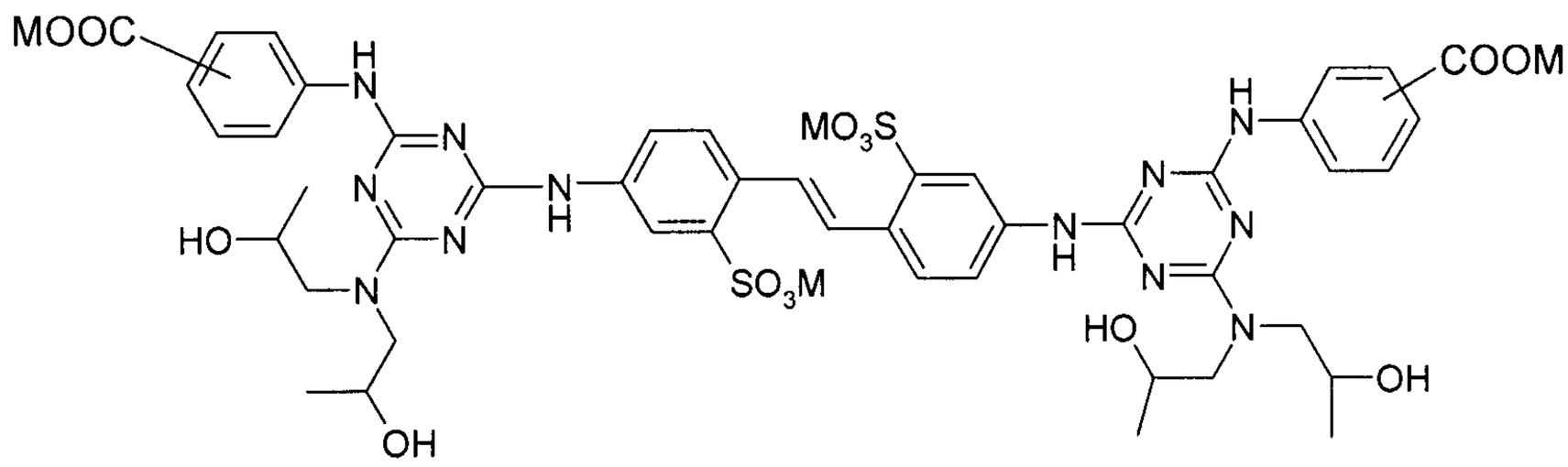
8. The use of the aqueous FWA preparation according to any one of claims 1 to 7, characterized in that the aqueous FWA preparation contains less than 5 % by weight of components other than components (a) and (b).

9. The use of the aqueous FWA preparation according to any one of claims 1 to 8, characterized in that the FWA has the following formula (Ia), wherein the carboxylic acid residues are, independently of each other, in *ortho*- or *para*–position



(Ia)

10. The use of the aqueous FWA preparation according to any one of claims 1 to 8, characterized in that the FWA has the following formula (Ib), wherein the carboxylic acid residues are, independently of each other, in *ortho*- or *para*-position



(Ib)

11. The use of the aqueous FWA preparation according to any one of claims 1 to 10, characterized in that the aqueous FWA preparation contains one FWA of formula (I).

12. The use of the aqueous FWA preparation according to any one of claims 1 to 10, characterized in that the aqueous FWA preparation contains two or three FWAs of formula (I).

13. The use of the aqueous FWA preparation according to any one of claims 1 to 12, for optically whitening paper or board in the pulp suspension or pulp.

14. The use of the aqueous FWA preparation according to claim 13, in wood-free pulp or wood-containing pulp.

15. The use of the aqueous FWA preparation according to any one of claims 1 to 12, for optically whitening paper at the surface.

16. A process for whitening paper comprising adding an aqueous FWA preparation as defined in any one of claims 1 to 12, to a pulp or pulp suspension, producing a paper sheet, and drying the sheet.

17. The process according to claim 16, characterized in that the aqueous FWA preparation is added, after dilution with water, to the pulp or pulp suspension.

18. Paper comprising the FWA as defined in any one of claims 1 to 12.

19. The paper according to claim 18, obtained by the process as defined in claim 16 or 17.

Fig. 1

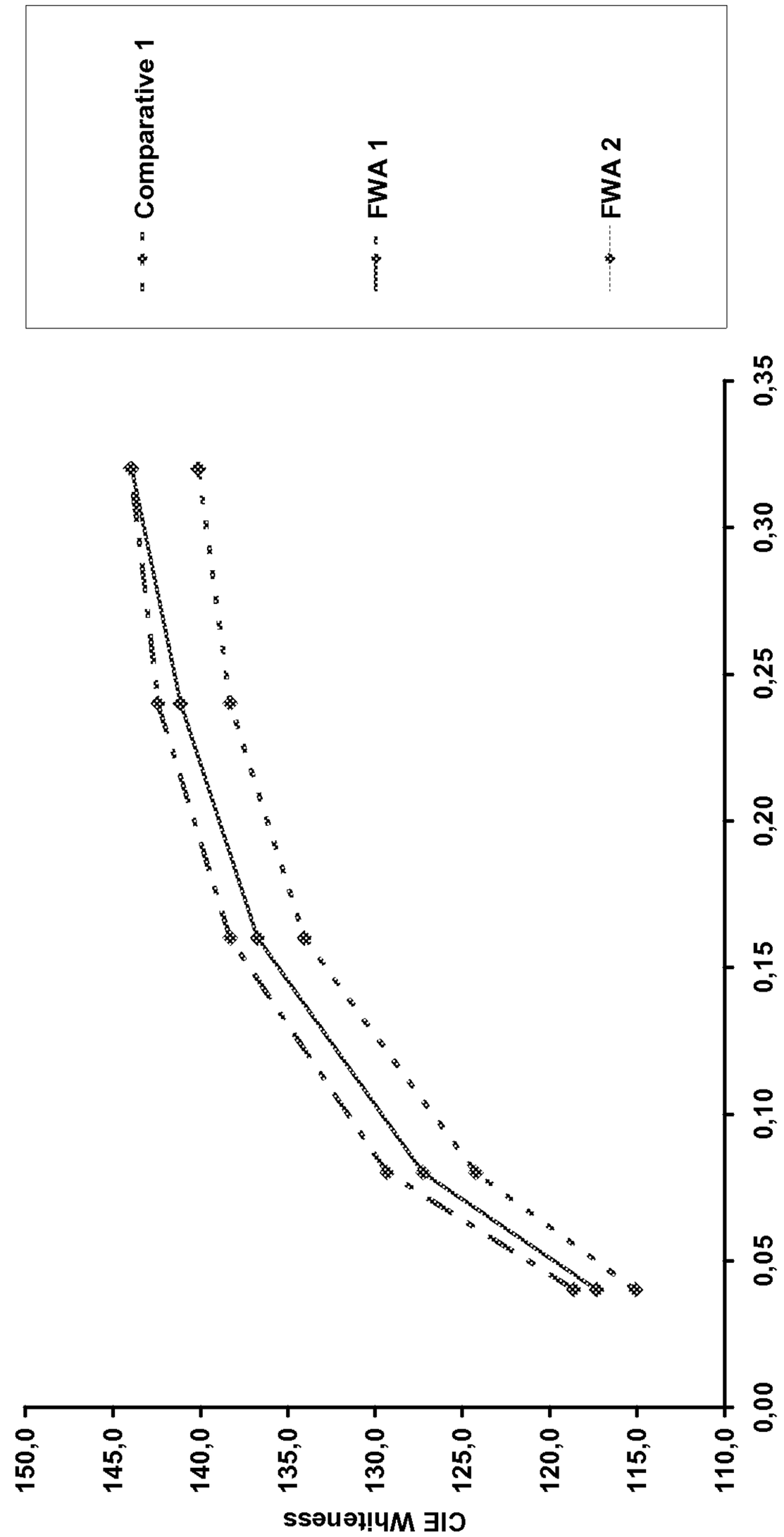
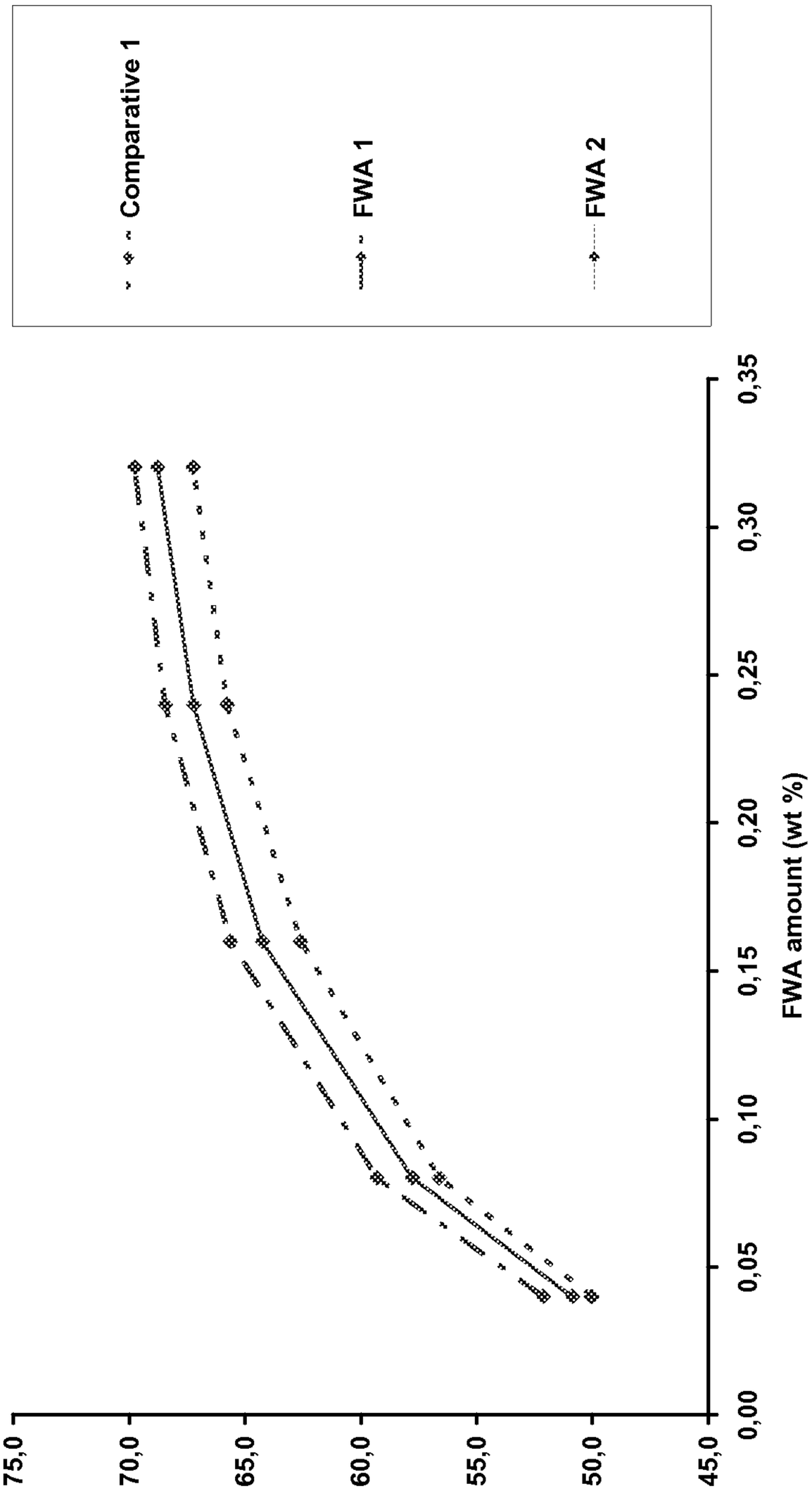

Comparative 1
FWA 1
FWA 2
CIE Whiteness
150,0
145,0
140,0
135,0
130,0
125,0
120,0
115,0
110,0
0,00
0,05
0,10
0,15
0,20
0,25
0,30
0,35
FWA amount (wt %)

Fig. 2


Comparative 1
FWA 1
FWA 2
CIE Whiteness
FWA amount (wt %)
75,0
70,0
65,0
60,0
55,0
50,0
45,0
0,00
0,05
0,10
0,15
0,20
0,25
0,30
0,35