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(54) **METHOD OF PRODUCING A DYED PAPER**

(57) A method for producing a mass dyed paper material by using food dyes comprises the following steps: adding a first fixing agent to a cellulose mixture being stirred; waiting for a first period of time; adding at least one food dye to the cellulose mixture being stirred; waiting for a second period of time; adding a second fixing agent to the cellulose mixture being stirred; waiting for a

third period of time; obtaining a dyed cellulose mixture; using the so obtained dyed cellulose mixture to form a sheet of mass dyed paper material. The aforesaid food dye is a solid food dye that is added as it is, or a solid food dye that is dissolved in a liquid formulation, or a solid food dye that is contained in a lacquer.

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Description

[0001] The invention relates to a method for producing a paper material, for example paper and cardboard, which is mass dyed, by using a food dye.

[0002] In the paper industry sector, known dyes are used to dye paper material (paper; cardboard) intended to come into contact with food products. More exactly, known dyes are used the specific use of which (dyeing of paper coming into contact with foodstuffs) is certified by certification bodies, such as for example the German ISEGA certification body.

[0003] Nevertheless, the aforesaid dyes, although they are approved by the main sector regulations, are not "food dyes". The latter, in Europe, are identified by a code comprising the letter "E" followed by a three-digit number (from 100 to 199). The possibility of using food dyes would be particularly desirable as it would confer an added value property to the paper material that is used in food packaging.

[0004] Furthermore, in terms of safety, the use of paper or cardboard that are dyed with a food dye (E100 - E199) could not be limited to the manufacture of food packaging. The use of food dyes could in fact be extended, for example, to the manufacture of materials intended for objects for school and/or play use or to the manufacture of the so-called "tissues", i.e. thin paper products such as toilet paper, paper handkerchiefs and paper for household use.

[0005] Known food dyes nevertheless have one drawback, which significantly limits the use thereof for dyeing a paper material. When a dye is added to the cellulose mixture, it is essential that the dye is fixed suitably to the cellulose fibres, so as to prevent the dye from being dispersed in the so-called under-cloth water. This would in fact be a problem for the treatment of the paper-mill waste water and would cause a non-homogeneous final colouring of the finished product (sheet of paper or cardboard).

[0006] The chemical nature of the food dyes is such as to give the latter a reduced affinity to the cellulose fibres. As a result, by using food dyes a paper material is obtained that is dyed in a not sufficiently homogeneous manner, which compromises the aesthetic appearance of the product and accordingly significantly reduces the commercial value thereof.

[0007] Furthermore, the lack of bond between cellulose fibres and dye causes a significant quantity of the latter to be dispersed in the under-cloth water, with a resulting increase in the environmental impact produced by the industrial process of paper dyeing.

[0008] Among the persons skilled in the art a strong need is therefore felt for a technical solution that enables the above disclosed drawbacks to be overcome.

Objects of the invention

[0009] One object of the invention is to improve the known methods for producing mass dyed paper materials.

[0010] Another object is to provide a method for producing a mass dyed paper material, in particular dyed paper and dyed cardboard, that enables food dyes to be used and an optimum interaction to be obtained between food dyes and cellulose fibres.

[0011] A further object is to provide a method for producing a mass dyed paper material, in particular dyed paper and dyed cardboard, which enables a homogeneously dyed paper material to be obtained by using food dyes.

[0012] Still another object is to provide a method for producing a mass dyed paper material, in particular dyed paper and dyed cardboard, which enables food dyes to be used and the environmental impact thereof to be reduced by limiting the discharge of dyes into the under-cloth water.

Short description of the invention

[0013] According to the invention, a method is provided for producing a mass dyed paper material, as defined in claim 1.

[0014] Owing to the invention, a method is made available for producing a mass dyed paper material, such as for example dyed paper or dyed cardboard, which enables the drawbacks of the prior art to be overcome.

[0015] In fact, in the method according to the invention, the used food dyes, together with specifically selected fixing agents, are added to a paper mixture in quantities and according to an order that are such as to suitably overcome the drawback of the poor affinity between food dyes and cellulose fibres. In this manner, it is possible to promote the bond between the food dyes and the cellulose fibres, thus obtaining a finished product (paper or cardboard) that is dyed in a suitably homogeneous manner and a good colour migration fastness of the paper in contact with water. It is further possible to reduce the environmental impact managing to obtain sufficiently clean under-cloth water.

Detailed description of the invention

[0016] A paper material, for example a sheet of paper, is "mass" dyed when the dyes are added inside the mixture during processing, this ensuring that the produced sheet is homogeneously dyed both internally and on the outer surfaces thereof (faces or sides of the sheet).

[0017] In order to produce a mass dyed paper material (paper or cardboard), the so-called mixture has to be first prepared, which is obtained from cellulose fibres. The cellulose fibres are mixed with water, pulped and refined using apparatuses of known type.

[0018] The mixture is prepared by setting and selecting various parameters: type of raw material that is used, degree of refinement of the mixture and concentration of the mixture.

[0019] The raw material can comprise: short cellulose fibres (for example, birch or eucalyptus cellulose), long cellulose fibres (for example, fir cellulose), cotton fibres, BCTMP (*bleached chemi-thermomechanical pulp*), recycled paper, etc.

[0020] The degree of refinement of the mixture is measured in °SR (Schopper-Riegler degrees) and influences the physical-mechanical properties of the paper material.

[0021] The concentration of the mixture is expressed as a mass percentage composition, i.e. as the percentage ratio between the mass of the fibres contained in the mixture and the total mass of the mixture. For example, a cellulose mixture having a mass equal to 500g, which is declared to have a 5% concentration of fibres, will contain 25g cellulose. Generally, in the paper mill, the mixture can vary from an initial 10-12% concentration to a 1% (or less) concentration, through several dilutions, as a function of the grammage that is desired for the paper or cardboard to be produced.

[0022] Once the mixture is produced, the latter can be added with chemical products (for example, dyes) that vary on the basis of the features that the finished product will have to have. Lastly, by using known apparatuses and methods, the sheet of paper is formed, pressed and subsequently dried.

[0023] In the following Table 1 a possible plant on an industrial scale (paper mill) for producing paper is schematically exemplified (from upstream to downstream):

Table 1

	Pulper for pulping the cellulose
	Dyeing vat
	Refining the mixture in the refiner
	Pre-machine chest
	Machine chest (dyed mixture reserve)
	Constant level chest (maintenance of constant feeding of mixture to the machine)
	Fan pump
	(addition of water to the mixture coming from the constant level box and delivery to the selector via the fan pump)
	Selector (sort of filtration of the mixture)
	Headbox (distribution of the mixture on the cloth)
	Flat table (formation of the sheet on the cloth and drainage of the water)
	Cylinder system (pressing and drying of the sheet)

[0024] The method according to the invention, conceived and tested experimentally by the Applicant, comprises the following steps:

- Adding a first fixing agent to a cellulose mixture being stirred;
- Waiting for a first period of time;
- Adding at least one food dye to the mixture being stirred;
- Waiting for a second period of time;
- Adding a second fixing agent to the mixture being stirred;
- Waiting for a third period of time;
- Obtaining a dyed cellulose mixture;
- Using the so obtained dyed cellulose mixture to form a sheet of a mass dyed paper material.

[0025] The first fixing agent and the second fixing agent can be the same as or different from one another. For example, using the E102 food dye, it is advisable that the first fixing agent is different from the second fixing agent.

[0026] The first fixing agent and the second fixing agent are added in predetermined w/w percentages, both expressed with respect to the w/w percentage of added food dye. The w/w percentages of the first fixing agent and of the second fixing agent vary on the basis of the type and concentration of dye that is used in the mixture.

[0027] The food dye is added in a predetermined w/w percentage, which is expressed with respect to the total quantity

of fibres contained in the mixture. The w/w percentage of dye to be used depends on the colouring intensity that it is desired to obtain and, as such, can be easily determined by the person skilled in the art.

[0028] In one embodiment of the invention, the w/w percentage of the first fixing agent and the w/w percentage of the second fixing agent are both comprised between 28% and 87% with respect to the w/w percentage of used dye.

[0029] The following Table shows, in correspondence with the type of dye, the quantity of each fixing agent to be used, expressed with a w/w % range with respect to the w/w percentage of used dye.

Table 2

Type of dye	First fixing agent (w/w %)	Second fixing agent (w/w %)
E102	64-87	64-87
E104	32-49	32-49
E110	51-69	51-69
E122	32-49	32-49
E124	43-58	43-58
E129	32-49	32-49
E132	51-75	51-75
E151	28-42	28-42

[0030] With reference to Table 2, to dye the mixture (for example) with the E122 dye, the w/w percentage of both a first fixing agent and of a second fixing agent to be used is comprised between 32% and 49% with respect to the w/w percentage of the dye. If, for example, the E122 dye is used (namely, added) in a percentage that is equal to 2% (with respect to the total quantity of fibres contained in the mixture), the percentage of the fixing agents to be used (namely, to be added) with respect to the total quantity of fibres contained in the mixture can be calculated via the proportions set out below.

[0031] For the lower limit of the reference range (w/w %):

$$32:100 = x: 2; \text{ from which } x = (2 \cdot 32)/100 = 0.64.$$

[0032] For the upper limit of the reference range (w/w %):

$$49:100 = x: 2; \text{ from which } x = (2 \cdot 49)/100 = 0.98.$$

[0033] From what has been disclosed above it is clear that - both for the first and the second fixing agent - a concentration comprised between 0.64% and 0.98% will have to be used with respect to the total quantity of fibres contained in the mixture. Once the above percentage range of concentrations has been calculated, the selection of the suitable percentage (of fixing agent) within the range can be effected autonomously by the person skilled in the art, since the quantities of fixing agents to be used can vary on the basis, for example, of the used mixture, of the degree of refinement of the mixture and of other parameters that depend on any particular productive process.

[0034] In one embodiment of the invention, the w/w percentage (with respect to the total quantity of fibres contained in the mixture) of the food dye is comprised between 0.01% and 7.5%.

[0035] In the context of the present description, as well as of the attached claims, the term "food dye" refers to the so-called "active" food dye, namely to a solid food dye (powder or granules) that is used as it is, or to a solid food dye (powder or granules) that is dissolved in a liquid formulation (namely, solid dye dissolved in a solvent), or to a solid food dye (powder or granules) that is contained in a lacquer.

[0036] In the context of the present description, as well as of the attached claims, the w/w percentage of a food dye that is added to the mixture thus corresponds to the w/w percentage of the solid food dye, which can be added as it is (namely, in solid form), or dissolved in a liquid formulation, or contained in a lacquer.

[0037] As it will be clear to a person skilled in the art, supposing for example to use a liquid formulation of a food dye (for example E122) containing 20% of dye in solid form (powder) and 80% of solvent (water), in order to carry out the

method according to the invention and adding, for example, 2 % w/w of dye (with respect to the total quantity of fibres contained in the mixture), 10% of the aforesaid liquid formulation will have to be used.

[0038] In one embodiment of the invention, the first period of time and the third period of time are at least 5 minutes, whereas the second period of time is at least 10 minutes.

[0039] In one embodiment of the invention, the food dye comprises a dye in powder or granules.

[0040] In another embodiment of the invention, the food dye comprises a liquid - namely solubilized (dissolved in a liquid formulation) - dye.

[0041] In a further embodiment of the invention, the food dye comprises a lacquer dye (dye in the insoluble form thereof), such as for example E 102.

[0042] By way of non-limiting example, the following Table 3 shows a list of known food dyes that are usable in the method according to the invention:

Table 3

E 102 - Tartrazine - Food yellow 4 - Colour Index 19140
E104 - Quinoline yellow - Food yellow 13 - Colour Index 47005
E110 - Sunset yellow FCF - Food yellow 3 - orange yellow S - Colour Index 15985
E122 - Azorubine - Carmoisine - Food red 3 - Colour Index 14720
E124 - Ponceau 4R - Cochineal red A - Food red 7 - new coccine - Colour Index 16255
E129 - Allura Red AC - Food red 17 - Colour index 16035
E132- Indigotin - Indigo carmine - Food blue 1- Colour index 73015
E151 - Brilliant Black BN - Black PN - Food black 1 - Colour Index 28440

[0043] The dyes of Table 3 are usable alone or in a mixture in the method according to the invention.

[0044] In one embodiment of the invention, the first fixing agent and the second fixing agent comprise: linear or branched cationic polyamines (such as for example: copolymer of epichlorohydrin and dimethylamine, copolymer of epichlorohydrin, dimethylamine and N-methylmethanamine; polymer of 1,2-ethanediamine with 2-(chloromethyl) oxirane and N-methylmethanamine; cyanoguanidine polymer with ammonium chloride and formaldehyde; polydiallyldimethylammonium chloride (polyDADMAC); poly(acrylamide-co-polydiallyldimethylammonium chloride).

[0045] In another embodiment of the invention, a predetermined w/w percentage of an auxiliary agent is added before adding the second fixing agent, where the aforesaid w/w percentage is expressed with respect to the total quantity of fibres contained in the mixture. The auxiliary agent can enable cleaner under-cloth water to be obtained and can partially cooperate in dyeing the mixture, but is not indispensable for carrying out the method according to the invention.

[0046] The auxiliary agent can be selected on the basis of the type of finished product to be obtained. For example, for the so-called "tissue" (toilet paper, paper handkerchiefs and paper for household use) aluminium sulphate or aluminium polychloride (PAC) are usable, whereas for the so-called "fine paper" (printing and writing paper) aluminium polychloride and alkyl ketene dimer (AKD) are usable.

[0047] In one embodiment of the invention, the auxiliary agent comprises aluminium sulphate and the corresponding w/w percentage is comprised between 0.2% and 0.9%.

[0048] In another embodiment of the invention, the auxiliary agent comprises aluminium polychloride and the w/w percentage is comprised between 0.1% and 0.5%.

[0049] In a further embodiment of the invention, the auxiliary agent comprises alkyl ketene dimer and the w/w percentage is comprised between 0.5% and 2%.

[0050] In a still further embodiment of the invention, the auxiliary agent comprises hexanedioic acid polymer with chloromethyl oxirane and N-(2-aminoethyl)-1,2-diaminoethane and the w/w percentage is comprised between 0.5% and 2%.

[0051] By way of non-limiting example of the invention, a procedure is disclosed below for producing mass dyed paper by the method according to the invention (Example 1).

Example 1 - Production of mass dyed paper by the method according to the invention

[0052] The Applicant has replicated - on the laboratory scale - a known procedure for the industrial production of paper, by applying the method according to the invention for dyeing the mixture.

[0053] A cellulose mixture was used having the properties shown in the following Table 4:

Table 4

Type of cellulose	50% short fibre cellulose + 50% long fibre cellulose
% cellulose in the mixture	5
Degree of refinement	28°SR
Water hardness	20°F
pH	7

[0054] 500 g of the aforesaid mixture were removed and placed in a receptacle of suitable capacity. By keeping the mixture stirred through instruments of known type, 213 mg of the first fixing agent, namely copolymer of epichlorohydrin and dimethylamine, were added. After 5 minutes, 500 mg of solubilized E129 dye were added. After 10 minutes, 50 mg of aluminium sulphate and 213 mg of the second fixing agent, namely copolymer of epichlorohydrin and dimethylamine, were added in rapid succession. After 5 minutes, the sheet-forming step started.

[0055] In order to form the sheet, the so-called sheet former was used. This instrument is composed of different parts: a column formed by an upper graduated transparent container and a lower part comprising a water discharging unit. Between the two parts, there is a frame with a filtering net. The sheet former is connected to the mains water supply and to a vacuum pump. The drying system, which is composed of a suitable plate, is placed next to the column.

[0056] Inside the graduated container of the column, the mains water and the mixture were added, then stirred. At the end of stirring the drainage process was started: the water moved from the graduated container to the part below the column, thus enabling the paper to be formed.

[0057] After which, the still wet sheet was removed and placed in a dryer for the time necessary for the drying thereof. At the end of drying, a hand-made sheet of paper was obtained.

[0058] By using the above disclosed procedure, it was possible to obtain a homogeneously mass dyed paper sheet, with a reduced loss of dye in the under-cloth water.

[0059] From what has been disclosed previously, it can be stated that the method according to the invention enables the predetermined objects to be reached. In fact, by the method according to the invention it is possible to mass dye paper materials in an optimum manner, in particular paper and cardboard, also using food dyes. This enables suitable conditions to be reached both for the dyed paper materials to be used in the food packaging and for dyed paper products intended for school and/or play use, as well as for thin paper products such as toilet paper, paper handkerchiefs and paper for household use.

[0060] It should also be noted that the method according to the invention enables the impact of the dyes on the environment and on the human organism to be reduced.

[0061] The dyes that are used by the method according to the invention - namely the food dyes - are such as to ensure a good environmental safety profile also at the end of life of the dye/finished product. In fact, the available PNEC (*predicted no-effect concentration*) values, LC50 96/h (fish acute toxicity test) and EC50 48/h (invertebrate acute toxicity test), together with the absence of metals linked to the dye molecule, enable a minimum impact for both the marine and land environment to be produced.

[0062] As the method according to the invention promotes the interaction between dye and fibres of the mixture, a suitable dyeing of the paper and a good quality of the under-cloth water - namely under-cloth water in which the dispersion of dye is minimized - are obtained contextually. Moreover, this result is also obtained owing to the selected % ranges for the fixing agents (see Table 2).

[0063] The dyes used by the method according to the invention are further such as to ensure the safety of the end user (human subject) also in the case of accidental contact and/or swallowing.

[0064] In fact, the used food dyes belong to the range of the food dyes that are authorized for use in human diet. Such dyes, according to the data present in bibliography (Sax's Dangerous Properties Of Industrial Materials, 11th edition - European Chemical Agency, Information on Chemicals Database), have values of oral LD50 that exceed on average 1000 mg/kg/day. This datum thus justifies a substantial "non-toxicity" condition of the paper finished product in the event of accidental swallowing, which condition is further supported by the low concentrations of dye (0.01 % - 7.5 %) that are requested by the method according to the invention for dyeing the mixture. Also an accidental and/or prolonged contact with the skin and/or the mucosae does not have foreseeable risks for the end user, as none of the used dyes shows sensitizing and/or irritating properties for the skin, eyes and/or mucosae.

[0065] Variations on and/or additions to what has been disclosed above are possible.

[0066] For example, although the method according to the invention has been disclosed through an example on the laboratory scale, the person skilled in the art is able to appropriately select suitable apparatuses and suitable quantities of raw materials and additives for carrying out the method according to the invention on an industrial scale. Moreover,

the method according to the invention can be combined with other methods that are known to the person skilled in the art and are used for dyeing paper material, for example surface dyeing methods.

Claims

1. Method for producing a mass dyed paper material, comprising the following steps:

- Adding a first fixing agent to a cellulose mixture being stirred;
- Waiting for a first period of time;
- Adding at least one food dye to said cellulose mixture being stirred;
- Waiting for a second period of time;
- Adding a second fixing agent to said cellulose mixture being stirred;
- Waiting for a third period of time;
- Obtaining a dyed cellulose mixture;
- Forming a sheet of said mass dyed paper material from said dyed cellulose mixture.

2. Method according to claim 1, wherein said food dye is a solid food dye and the form in which said solid food dye is added is selected from the group consisting of: solid food dye as it is, solid food dye dissolved in a liquid formulation and solid food dye contained in a lacquer.

3. Method according to claim 1 or 2, wherein said food dye is added in a predetermined w/w percentage, said w/w percentage being expressed with respect to the total quantity of fibres contained in said mixture, and wherein said first fixing agent and said second fixing agent are added in predetermined w/w percentages, said w/w percentages being expressed with respect to said w/w percentage of said food dye.

4. Method according to claim 3, wherein said predetermined w/w percentage of said food dye is comprised between 0.01% and 7.5%.

5. Method according to claim 3 or 4, wherein said predetermined w/w percentage of said first fixing agent and said predetermined w/w percentage of said second fixing agent are comprised between 28% and 87%.

6. Method according to any one of claims 1 to 5, wherein said first fixing agent and said second fixing agent are selected from the group consisting of: linear cationic polyamines, branched cationic polyamines, polydiallyldimethylammonium chloride and poly (acrylamide-co-polydiallyldimethylammonium chloride).

7. Method according to claim 6, wherein said cationic polyamines are selected from the group consisting of: copolymer of epichlorohydrin and dimethylamine; copolymer of epichlorohydrin, dimethylamine and N-methylmethanamine; polymer of 1,2-ethanediamine with 2-(chloromethyl) oxirane and N-methylmethanamine; cyanoguanidine polymer with ammonium chloride and formaldehyde.

8. Method according to any one of claims 1 to 7, wherein said first fixing agent is different from said second fixing agent.

9. Method according to any one of claims 1 to 8, wherein said first period of time and said third period of time are at least 5 minutes each, while said second period of time is at least 10 minutes.

10. Method according to any one of claims 1 to 9, further comprising adding a predetermined w/w percentage of an auxiliary agent to said cellulose mixture being stirred, said predetermined w/w percentage being expressed with respect to the total quantity of fibres contained in said mixture.

11. Method according to claim 10, wherein said adding said auxiliary agent is carried out before said adding said second fixing agent.

12. Method according to claim 10 or 11, wherein said auxiliary agent is selected from the group consisting of: aluminium sulphate; aluminium polychloride; alkyl ketene dimer; hexanedioic acid polymer with chloromethyl oxirane and N-(2-aminoethyl)-1,2-diaminoethane.

13. Method according to claim 12, wherein said predetermined w/w percentage of said auxiliary agent is expressed by

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the following percentage ranges:

5	Aluminium sulphate	0.2% - 0.9%
	Aluminium polychloride	0.1% - 0.5%
	Alkyl ketene dimer	0.5% - 2.0%
	Hexanedioic acid polymer with chloromethyl oxirane and N-(2-aminoethyl)-1,2-diaminoethane	0.5% - 2.0%.

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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

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