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(12) (19) (CA) **Demande-Application**

(21) (A1) **2,296,448**
(86) 1998/04/06
(87) 1999/04/08

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(51) Int.Cl.^{7} D21H 23/24, B41M 5/00
(30) 1997/09/30 (60/060,390) US

(54) PROCEDE DE COUCHAGE DE PAPIER A L'AIDE DE 2-OXETANONE ET DE COLOPHANE ET PAPIER AINSI PREPARE

(54) METHOD FOR SURFACE SIZING PAPER WITH 2-OXETANONE AND ROSIN AND PAPER PREPARED THEREBY

(57) Procédé de couchage de papier par ajout sur la surface dudit papier d'une composition de couchage comportant une colophane et une 2-oxétanone. Le papier ainsi couché offre une meilleure performance en cas d'impression à jet d'encre que le papier identique mais pour lequel la composition de couchage contient uniquement de la colophane ou uniquement de la 2-oxétanone, lorsque l'impression est évaluée en tenant compte d'au moins une propriété choisie parmi la densité optique, les bavures, l'effet de mèche, la précision des limites et le déteintage. Le papier ainsi couché présente une meilleure adhérence du toner, un coefficient de frottement plus élevé et une largeur de bande de coefficient de frottement plus faible que le papier identique mais dont la composition de couchage ne contient que de la 2-oxétanone. Ledit papier est également capable d'obtenir des bons résultats dans des analyses destinées à mesurer sa transformabilité sur un équipement de transformation correspondant à l'état le plus récent de la technique et ses performances sur des machines d'utilisation finale fonctionnant à grande vitesse.

(57) There is disclosed a method for sizing paper by adding to the surface of the paper a sizing composition comprising a rosin and a 2-oxetanone. The sized paper performs better in ink jet printing than does paper that is the same except that the size composition contains only rosin or only 2-oxetanone, when the printing is evaluated for at least one property selected from the group consisting of optical density, feathering, wicking, edge roughness and bleed. The sized paper also has higher toner adhesion, a higher coefficient of friction and a lower coefficient of friction bandwidth than does paper that is the same except that the size composition contains only 2-oxetanone. The paper is also capable of performing effectively in tests that measure its convertibility on state-of-the-art converting equipment and its performance on high speed end-use machinery.

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ^{6} :
D21H 23/24, B41M 5/00 // D21H 21:16, 17:71, 17:17, 17:28, 17:62, 17:66

A1

(11) International Publication Number: **WO 99/16975**

(43) International Publication Date: 8 April 1999 (08.04.99)

(21) International Application Number: PCT/US98/06803

(22) International Filing Date: 6 April 1998 (06.04.98)

(30) Priority Data:
60/060,390 30 September 1997 (30.09.97) US

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(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: METHOD FOR SURFACE SIZING PAPER WITH 2-OXETANONE AND ROSIN AND PAPER PREPARED THEREBY

(57) Abstract

There is disclosed a method for sizing paper by adding to the surface of the paper a sizing composition comprising a rosin and a 2-oxetanone. The sized paper performs better in ink jet printing than does paper that is the same except that the size composition contains only rosin or only 2-oxetanone, when the printing is evaluated for at least one property selected from the group consisting of optical density, feathering, wicking, edge roughness and bleed. The sized paper also has higher toner adhesion, a higher coefficient of friction and a lower coefficient of friction bandwidth than does paper that is the same except that the size composition contains only 2-oxetanone. The paper is also capable of performing effectively in tests that measure its convertibility on state-of-the-art converting equipment and its performance on high speed end-use machinery.

METHOD FOR SURFACE SIZING PAPER WITH 2-OXETANONE AND ROSIN AND PAPER PREPARED THEREBY

Field of the Invention

This invention relates to processes for surface sizing paper, to paper prepared by the processes, and to processes for preparing surfaces sizes.

Background of the Invention

Current applications for fine paper require particular attention to sizing before conversion or end-use, such as high speed photocopies, envelopes, forms bond, including computer paper, and adding machine paper. Paper is conventionally sized by addition of sizing agents to the "wet end" of the paper process (internal addition), i.e., to the pulp before sheet formation, or by addition of sizing agents to the surface of already formed paper sheet that has been at least partially dried (surface sizing).

Rosin sizes are widely used paper sizing agents. Although they are described in the literature as being useful for both internal and surface sizing, they are generally not used for surface sizing commercially. Alkyl ketene dimers display high sizing efficiency, but may cause problems in size reversion, toner adhesion and high speed paper converting. Variable coefficient of friction is at least one factor leading to the problems in high speed converting operations.

Recently non-solid 2-oxetanones such as alkenyl ketene dimers and ketene multimers have been described that are useful for internal and surface sizing and that overcome the deficiencies in high speed converting. These materials are disclosed in U.S. Patent Application Serial No. 08/601,113 (allowed), filed February 16, 1996, which is incorporated herein by reference in its entirety, and in U.S. Patent No. 5,685,815, which is incorporated herein by reference in its entirety. Precis®2000 and Precis®3000 sizing agents (available from Hercules Incorporated, Wilmington, DE) are examples of such sizes. They are widely used commercially for internal sizing, but not for surface sizing because they do not contribute to good toner adhesion and other surface properties.

Starch and other polymeric sizes are widely used for surface sizing. These materials generally exhibit improved toner adhesion, little or no effect on coefficient of friction, no effect, or an improved effect on high speed converting, and no size reversion when compared to alkyl ketene dimers; however, they are less efficient at sizing than the alkyl ketene dimers.

As a result of all of the above, most papers at the present time are internally sized with alkenylsuccinic anhydride, alkyl ketene dimers, alkenyl ketene dimers or rosin sizes.

There is a need to provide surface sizing agents that overcome the problems enumerated above, because there are substantial advantages to be gained from surface sizing when compared to internal sizing. Among them are:

a) Efficiency. The surface size components are completely retained in the system; whereas in internal sizing a significant amount is lost in the white water. Moreover, in those applications where only a surface response is needed, surface sizing permits keeping the majority of the size at the surface, thus gaining the maximum response for a minimum amount of material.

b) Environmental. The high retention of surface size components minimizes environmental contamination.

c) Fiber bonding. Sizes applied at the surface are less likely to interfere with fiber-fiber bonding because the bonds have already been formed when the sizes are applied.

Now it has been found unexpectedly that treatment of paper with a combination of 2-oxetanone not solid at 25°C and rosin size can produce paper with a unique balance of final properties that cannot be achieved by using either of the size types alone. The combination sizes provide paper that exhibits better water holdout than paper that is the same except that the sizing composition contains only rosin size. The combination size also provides paper that performs better in ink jet printing than does paper that is the same except that the size composition contains only 2-oxetanone not solid at 25°C or only rosin size. Furthermore, the paper exhibits better toner adhesion, higher coefficient of friction and a lower coefficient of friction bandwidth than does paper that is the same except that the size composition contains only 2-oxetanone. The paper is also capable of performing effectively in tests that measure its convertibility on state-of-the-art converting equipment and its performance on high speed end-use machinery.

U.S. Patent No. 4,522,686 to Dumas describes sizing compositions in the form of aqueous dispersions comprising cellulose reactive sizing agent such as ketene dimer, fortified rosin and a water-soluble, nitrogen-containing cationic dispersing agent. The compositions are described as highly efficient internal or surface sizes.

U.S. Patent No. 5,456,800 to Tansely et al. discloses internally sizing liquid packaging board utilizing an aqueous emulsion of cellulose reactive size, including alkyl ketene dimer, cellulose non-reactive size, including rosin derivatives.

U.S. Patent No. 5,510,003 to Colasurdo et al. relates to a method for internally sizing paper using an aqueous dispersion of sizing agents comprising a rosin component and a cellulose reactive size such as alkyl dimer.

Summary of the Invention

A process for sizing paper comprises a) providing an aqueous pulp suspension; b) sheeting and drying the aqueous pulp suspension to obtain paper; c) applying to the paper an aqueous size composition comprising at least one 2-oxetanone that is not solid at 25°C and at least one rosin; and d) drying the paper. Paper prepared by the process exhibits better water holdout than paper that is the same except that the sizing composition contains only rosin. The paper also performs better in ink jet printing than does paper that is the same except that the size composition contains only rosin or only 2-oxetanone that is not solid at 25°C when the printing is evaluated for at least one proerty selected from the group consisting of optical density, feathering, wicking, edge roughness and bleed. In addition, the paper has higher toner adhesion, has a higher static or kinetic coefficient of friction (COF), and has lower COF bandwidth than does paper that is the same except that the size composition contains only 2-oxetanone that is not solid at 25°C.

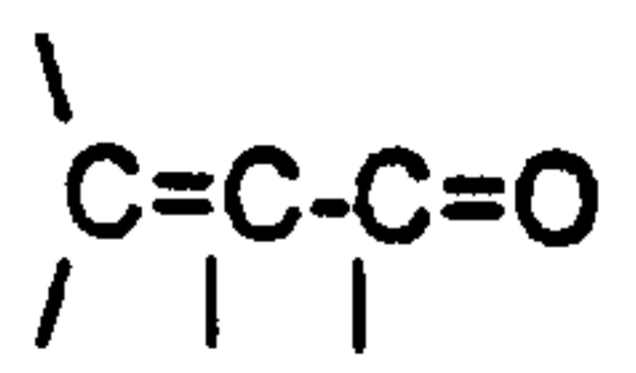
Detailed Description of the Invention

Rosin

The rosin useful for the present invention can be any modified or unmodified rosin suitable for sizing paper, including unfortified rosin, fortified rosin and extended rosin, as well as rosin esters, and mixtures and blends thereof.

The rosin used in this invention can be any of the commercially available types of rosin, such as wood rosin, gum rosin, tall oil rosin, and mixtures of any two or more, in their crude or refined state. Tall oil rosin and gum rosin are preferred. Partially hydrogenated rosins and polymerized rosins, as well as rosins that have been treated to inhibit crystallization, such as by heat treatment or reaction with formaldehyde, also can be employed.

A fortified rosin useful in this invention is the adduct reaction product of rosin and an acidic compound containing the

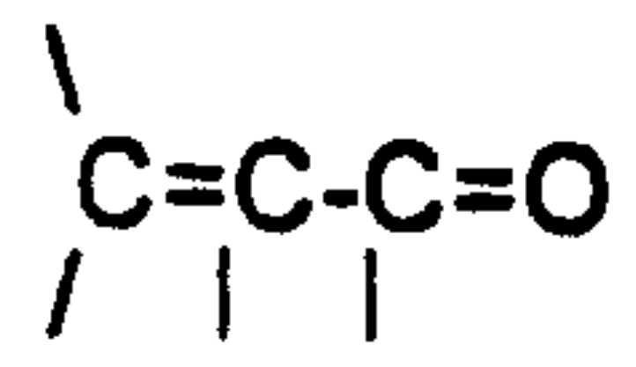


$$\begin{array}{l} \backslash \\ C=C-C=O \\ / \quad | \quad | \end{array}$$

group and is derived by reacting rosin and the acidic compound at elevated temperatures of from about 150°C to about 210°C.

The amount of acidic compound employed will be that amount which will provide fortified rosin containing from about 1% to about 16% by weight of adducted acidic compound based on the weight of the fortified rosin. Methods of preparing fortified rosin are well known to those skilled in the art. See, for example, the methods disclosed and described in U.S. Patents 2,628,918 and 2,684,300, the disclosures of which are incorporated herein by reference.

Examples of acidic compounds containing the



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group that can be used to prepare the fortified rosin include the α,β-unsaturated organic acids and their available anhydrides, specific examples of which include fumaric acid, maleic acid, acrylic acid, maleic anhydride, itaconic acid, itaconic anhydride, citraconic acid and citraconic anhydride. Mixtures of acids can be used to prepare the fortified rosin if desired. Thus, for example, a mixture of the acrylic acid adduct of rosin and the fumaric acid adduct can be used to prepare the novel dispersions of this invention. Also, fortified rosin that has been substantially completely hydrogenated after adduct formation can be used.

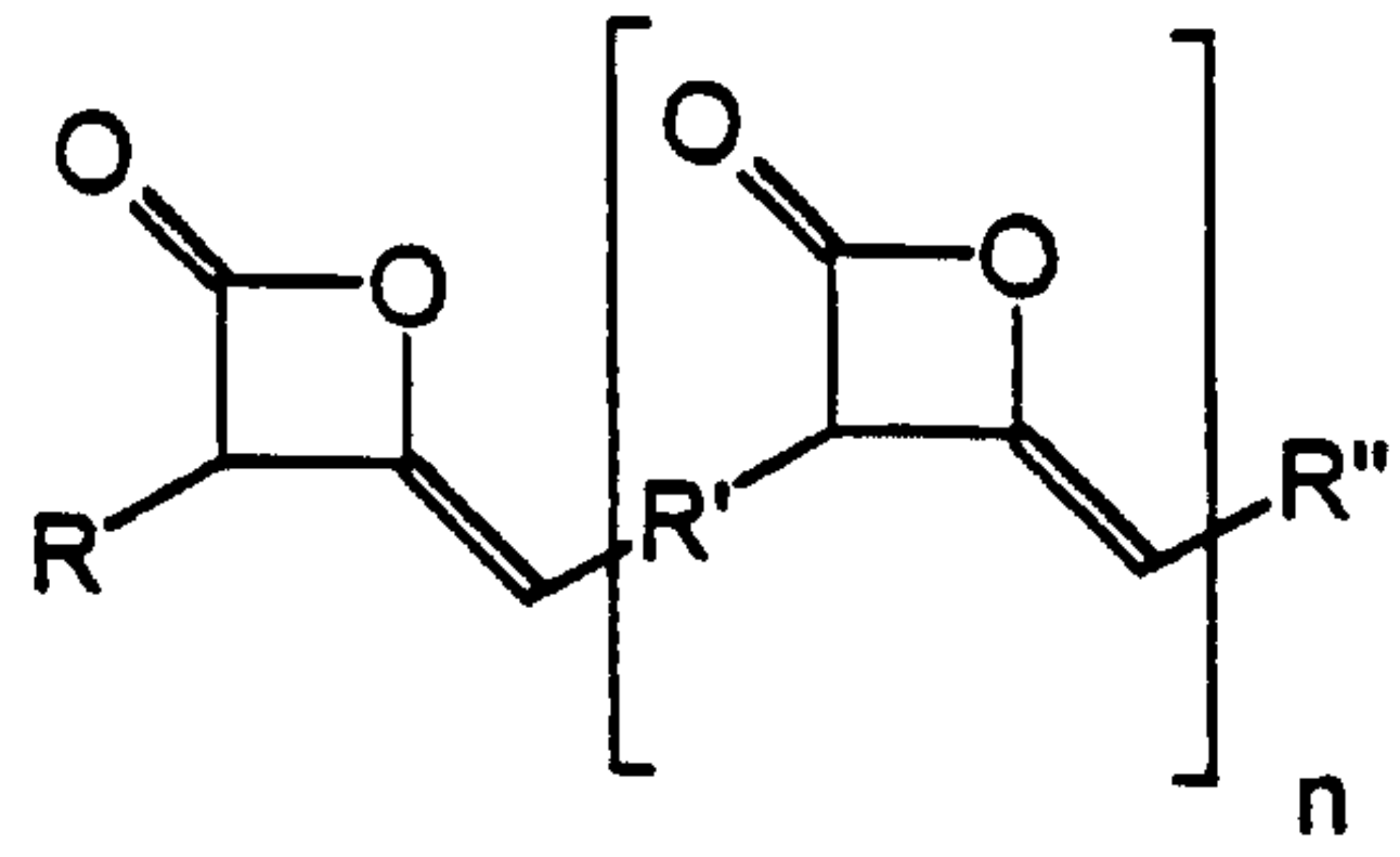
Various rosin esters of types well known to those skilled in the art can also be used in the present invention. Alcohols that can be used to prepare the various esters of rosin include, but are not restricted to glycerol, ethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, 1,4-butanediol, sorbitol and mannitol. Preferred alcohols are pentaerythritol and glycerol. A more preferred alcohol is pentaerythritol.

The unfortified or fortified rosin or rosin esters can be extended, if desired, by known extenders therefor such as waxes (particularly paraffin wax and microcrystalline wax), hydrocarbon resins including those derived from petroleum hydrocarbons and terpenes, and the like. This is accomplished by melt blending or solution blending with the rosin or fortified rosin from about 10% to about 100% by weight, based on the weight of rosin or fortified rosin, of the extender.

Blends of fortified rosin and unfortified rosin; and blends of fortified rosin, unfortified rosin, rosin esters and rosin extender can be used also. Blends of fortified and unfortified rosin may comprise, for example, about 25% to 95% fortified rosin and about 75% to 5% unfortified rosin. Blends of fortified rosin, unfortified rosin, and rosin extender may comprise, for example, about 5% to 45% fortified rosin, 0 to 50% rosin, and about 5% to 90% rosin extender.

2-Oxetanone

The 2-oxetanones for use in the invention are ketene dimers and multimers having the structure of formula 1


(1)

where n is an integer of 0 to about 20, preferably 0 to about 6, more preferably 0 to about 3, and most preferably 0, R and R", which may be the same or different, are saturated or unsaturated straight chain or branched alkyl groups having 6 to 24, preferably 10 to 20 and more preferably 14 to 16 carbon atoms; and R' is a saturated or unsaturated straight chain or branched alkyl group having from about 2 to about 40, preferably about 4 to about carbon atoms, and wherein at least 25% of the R and R" groups is unsaturated.

The liquid ketene dimers (n in formula 1 is 0) and multimers (n in formula 1 is an integer of 1 or greater) may comprise a mixture of ketene dimer or multimer compounds that are the reaction product of a reaction mixture comprising unsaturated monocarboxylic fatty acids. The reaction mixture may further comprise saturated monocarboxylic fatty acids and dicarboxylic acids. Preferably the reaction mixture for preparing the mixture of dimer or multimer compounds comprises at least 25 wt% unsaturated monocarboxylic fatty acids, and more preferably at least 70 wt% unsaturated monocarboxylic fatty acids.

The unsaturated monocarboxylic fatty acids included in the reaction mixture preferably have 10-26 carbon atoms, more preferably 14-22 carbon atoms, and most preferably 16-18 carbon atoms. These acids include, for example, oleic, linoleic, dodecenoic, tetradecenoic (myristoleic), hexadecenoic (palmitoleic), octadecadienoic (linolelaidic), octadecatrienoic (linolenic), eicosenoic (gadoleic), eicosatetraenoic (arachidonic), cis-13-docosenoic (erucic), trans-13-docosenoic (brassidic), and docosapentaenoic (clupanodonic) acids, and their acid halides, preferably chlorides. One or more of the monocarboxylic acids may be used. Preferred unsaturated monocarboxylic fatty acids are oleic, linoleic, linolenic and palmitoleic acids, and their acid halides. Most preferred unsaturated monocarboxylic fatty acids are oleic and linoleic acids, and their acid halides.

The saturated monocarboxylic fatty acids used to prepare the ketene dimer and multimer compounds used in this invention preferably have 10-26 carbon atoms, more preferably 14-22 carbon atoms, and most preferably 16-18 carbon atoms. These acids include, for example, stearic, isostearic, myristic, palmitic, margaric, pentadecanoic, decanoic, undecanoic, dodecanoic, tridecanoic, nonadecanoic, arachidic and behenic acids, and their halides, preferably chlorides. One or more of the saturated monocarboxylic fatty acids may be used. Preferred acids are palmitic and stearic.

The alkyl dicarboxylic acids used to prepare the ketene multimer compounds for use in this invention preferably have 6-44 carbon atoms, and more preferably 9-10, 22 or 36 carbon atoms. Such dicarboxylic acids include, for example, sebacic, azelaic, 1,10-dodecanedioic, suberic, brazylic, docosanedioic acids, and C_{36} dimer acids, e.g. EMPOL 1008 available from Henkel-Emery, Cincinnati, Ohio, U.S.A, and their halides, preferably chlorides. One or more of these dicarboxylic acids can be used. Dicarboxylic acids with 9-10 carbon atoms are more preferred. The most preferred dicarboxylic acids are sebacic and azelaic acids.

When dicarboxylic acids are used in the preparation of the ketene multimers for use in this invention, the maximum mole ratio of dicarboxylic acid to monocarboxylic acid (the sum of both saturated and unsaturated) is preferably about 5. A more preferred maximum is about 4, and the most preferred maximum is about 2. The mixture of dimer and multimer compounds may be prepared using methods known for the preparation of standard ketene dimers. In the first step, acid halides, preferably, acid chlorides, are formed from a mixture of fatty acids, or a mixture of fatty acids and dicarboxylic acid, using PCl_3 or another halogenating, preferably chlorinating, agent. The acid halides are then converted to ketenes in the presence of tertiary amines (including trialkyl amines and cyclic alkyl amines), preferably triethylamine. The ketene moieties then dimerize to form the desired compounds.

2-Oxetanones not solid at 25°C are available as Precis® sizing agents from Hercules Incorporated, Wilmington, Delaware.

The rosins and 2-oxetanones will generally be used as aqueous emulsions or dispersions. The dispersions of the aqueous size compositions may be prepared by mixing dispersions of the separate components, or alternatively, by dispersing blends of rosin and 2-oxetanone. Mixing dispersions of the two components is the preferred method. The mixing may take place at the size press by adding separate dispersion components to the size press, or it may take place prior to use at the size press. In this regard, it is an advantage of the invention that the premixed dispersions have good storage stability, e.g. no substantial separation or formation of solids, and maintain their ability to be used for sizing for greater than eight days at room temperature. Preferably the premixed dispersions have good storage stability for greater than about 20 days, more preferably greater than about 60 days and most preferably greater than about 180 days.

The aqueous pulp suspension of step (a) of the process is obtained by means well known in the art, such as known mechanical, chemical and semichemical, etc., pulping processes. Normally, after the mechanical grinding and/or chemical pulping step, the pulp is washed to remove residual pulping chemicals and solubilized wood components. Either bleached or unbleached pulp fiber may be utilized in the process of this invention. Recycled pulp fibers are also suitable for use.

The sheeting and drying of the pulp suspension is also carried out by methods well known in the art. There is a variety of materials which in the commercial practice of making paper are commonly add to the aqueous pulp suspension before it is converted into paper, and may be used in the instant process as well. These include, but are not restricted to, wet strength resins, internal sizes, dry strength resins, retention aids, alum, fillers, pigments and dyes.

Paper sized by processes of this invention is commonly known as surface sized paper. Preferably, in surface sizing processes, the size is applied to the surface of the paper from a size press, which can be any type of coating or spraying equipment, but most commonly is a puddle, gate roller or metered blade type of size press.

Paper coatings are also applied to the surface of paper, but they are completely different in function and composition from surface sizes. Paper coating compositions have much higher viscosities than surface size compositions, and thus cannot readily be applied by a size press on a typical paper machine. Paper coatings contain pigment at levels 3 to 20 times higher than that of polymeric binder; whereas in a typical surface size, pigments are optional. Preferably, they are used at levels of 0 to about 50% by weight, more preferably 0 to 30 % by weight of the total solids level of the aqueous size composition.

For the sizing processes of the invention, the sizing composition is preferably mixed with a solution of starch or starch derivative prior to its application to the paper. The starch may be of any type, including but not limited to oxidized, ethylated, cationic and pearl starch, and is preferably used in aqueous solution. The typical size press starch solution preferably contains a minimum of about 1% by weight starch in water, with a pH between about 6 and 9. A more preferable minimum starch level is about 2%, and the most preferable about 3%. The preferred maximum level of starch in the size composition is about 20% by weight. A more preferable maximum is about 16% and the most preferable about 12% by weight. Small amounts of other additives may be present as well, e.g., optical brighteners and defoamers. The amount of size composition added to the starch solution to form the size press compound is such that the minimum total of rosin and 2-oxetanone solids level in the final size press compound is preferably about 0.01 wt.% based on the total weight of the size composition. A more preferable minimum is about 0.02 wt.%. The preferred maximum total of rosin and 2-oxetanone solids level in the final size press compound is preferably about 2 wt.% and more preferably about 1 wt.%.

The ratio, on a dry basis, of rosin to 2-oxetanone in the aqueous size compositions preferably has a minimum value of about 0.2:1. More preferably the minimum is about 0.5:1, and most preferably about 1:1. The maximum ratio is preferably about 50:1, more preferably about 40:1 and most preferably about 30:1.

The amount of surface size applied at the size press is such as to provide starch at a preferable minimum level of about 1 wt.% on a dry basis based on the dry weight of the paper. A more preferable minimum level is about 2 wt.%, and a most preferable minimum level about 3 wt.%. The maximum level of starch applied is preferably about 8 wt.%, more preferably about 7 wt.% and most preferably about 6 wt.% on a dry basis based on the dry weight of the paper.

Preferably the surface size is applied at the size press in an amount to provide a minimum amount of size composition, i.e. total of rosin and 2-oxetanone of about 0.01 wt.% on a dry basis based on the dry weight of the paper. A more preferable minimum amount is about 0.03 wt.%, and a most preferable minimum amount about 0.05 wt.%. Preferably the maximum amount of size composition will be about 1 wt.%, more preferably about 0.7 wt.% and most preferably about 0.5 wt.% on a dry basis based on the dry weight of the paper.

The amount of surface size applied will also provide a minimum amount of 2-oxetanone of about 0.005 wt.% on a dry basis based on the dry weight of the paper. A more preferable minimum amount is about 0.01 wt.%, and a most preferable minimum amount is about 0.02 wt.%. Preferably the maximum amount of 2-oxetanone applied is about 0.5 wt.%, more preferably about 0.3 wt.%, and most preferably about 0.2 wt.% on a dry basis based on the dry weight of the paper.

The amount of surface size applied will also provide a minimum amount of rosin of about 0.01 wt.% on a dry basis based on the dry weight of the paper. A more preferable minimum amount is about 0.02 wt.%, and a most preferable minimum amount is about 0.04 wt.%. Preferably the maximum amount of rosin applied is about 0.5 wt.%, more preferably about 0.4 wt.%, and most preferably about 0.3 wt.% on a dry basis based on the dry weight of the paper.

After application of the surface size, the sheets are dried utilizing any of the conventional drying procedures well known in the art.

One advantage of the processes of this invention is that internal sizing is not needed. However in some situations it is desirable to internally size, because internal sizing helps prevent the surface size from soaking into the sheet, thus allowing it to remain on the surface where it has maximum effectiveness. The paper to be surface sized by the processes of this invention may also be internally sized by addition of sizing agents to the pulp suspension before it is converted to a paper sheet.

The internal sizing agents encompass any of those commonly used at the wet end of a fine paper machine. These include rosin sizes, fortified rosin sizes, ketene dimers and multimers, and alkenylsuccinic anhydrides. The rosin and 2-oxetanone sizes disclosed herein for surface sizing may also be used for internal sizing. The internal sizes are preferably used at levels of from about 0.05 wt.% to about 0.3 wt.% on a dry basis based on the weight of the dry paper sheet. More preferable levels are from about 0.01 to about 0.2 wt.%, and the most preferable levels from about 0.01 to about 0.1 wt.%.

Methods and materials utilized for internal sizing with rosin are discussed by E. Strazdins, with alkyl ketene dimer by R.E. Cates, D.H. Dumas and D.B. Evans, and with alkenyl succinic anhydride by C.E. Farley and R.B. Wasser all in *The Sizing of Paper, Second Edition*, edited by W.F. Reynolds, Tappi Press, 1989, pages 1-62.

Suitable ketene dimers and multimers for internal sizing are the same as those discussed above in connection with cellulose reactive sizes.

Another benefit of the invention is that paper produced by the processes of the invention has unique properties not obtained by using either rosin or 2-oxetanone sizes alone. In general these properties combine the high efficiency of 2-oxetanone sizes with improved toner adhesion, ability to use the paper in high speed converting or reprographic operations, good balance of color and black ink jet printing, no size reversion, and no reduction of coefficient of friction as is often associated with 2-oxetanone sizes.

Specifically, the sized paper of this invention performs better in ink jet printing than does paper that is the same except that the size composition contains only 2-oxetanone size, when the printing is evaluated for at least one property selected from the group consisting of optical density, feathering, wicking, edge roughness and bleed. The sized paper also has a higher coefficient of friction and a lower coefficient of friction bandwidth than does paper that is the same except that the size composition contains only 2-oxetanone size. Bandwidth is defined as the difference between the average maximum and average minimum of the stick-slip response in the kinetic coefficient of friction curve.

When the surface sized paper of this invention is to be used for ink jet printing it has been found that the quality of the ink jet printing is enhanced by including in the surface size composition various salts of cationic metal ions that are soluble in water at about pH 7 to about pH 9. Examples of salts which are effective for this use are sodium chloride, sodium sulfate, calcium chloride, calcium bromide, magnesium chloride, magnesium bromide, aluminum sulfate and poly aluminum chloride. Preferred salts are calcium chloride, calcium bromide, magnesium chloride and magnesium bromide. More preferred salts are calcium and magnesium chlorides. The weight ratio of the salt to other solids contained in the size composition is from about 1:20

to about 20:1. More preferably the ratios are about 1:5 to about 5:1, and most preferably about 1:3 to about 3:1.

The paper of this invention is also capable of performing effectively in tests that measure its convertibility on state-of-the-art converting equipment and its performance on high speed end-use machinery. In particular, the paper according to the invention that can be made into folded continuous forms bond having a basis weight of about 48.7 to 95.5 g/ m^2 (30 to 60 lb/3000 ft$^{2)}$, preferably about 64.5 to 81.0 g/ m^2 (40 to 50 lb/3000 ft^2), is capable of running on an IBM Model 3800 high speed, continuous-forms laser printer without causing billowing in the cooling section (after the fuser section and before the take-up section) of greater than about 12.7 cm (5 inches), preferably 7.6 cm (3 inches) or less, after ten minutes running time.

Further, the preferred paper according to the invention, that can be made into sheets of 21.6 cm x 28 cm (8 1/2 x 11 inch) reprographic cut paper having a basis weight of about 56.1 to 90.0 g/m^2 (15-24 lb/1300 ft^2) is capable of running on a IBM model 3825 high-speed copier without causing misfeeds or jams at a rate of 5 or less in 10,000, preferably at a rate of 1 or less in 10,000. By comparison, paper sized with standard alkyl ketene dimer has a much higher rate of double feeds on the IBM 3825 high speed copier (14 double feeds in 14,250 sheets). In conventional copy-machine operation, 10 double feeds in 10,000 is unacceptable. A machine manufacturer considers 1 double feed in 10,000 sheets to be unacceptable.

The paper of this invention in the form of a roll of continuous forms bond paper having a basis weight of about 32.6 to 39.1 g/m^2 (20-24 lb/3000 ft^2) can be converted to a standard perforated continuous form on a Hamilton-Stevens continuous forms press at a press speed of at least about 541 m (1775 feet) per minute, preferably at least about 579 m (1900 feet) per minute.

The paper of this invention can also be made into a roll of envelope paper having a basis weight of about 75.2 to 90.1 g/m^2 (20-24 lb/1300 ft^2) that can be converted into at least about 900 envelopes per minute, preferably at least about 1000 per minute on a Winkler & Dunnebier CH envelope folder.

The paper of this invention can be run at a speed of at least about 58 sheets per minute on a high speed IBM 3825 sheet-fed copier with less than 1 in 10,000 double feeds or jams.

This invention is illustrated by the following examples, which are exemplary only and not intended to be limiting. All percentages, parts, etc., are by weight, based on the weight of the dry pulp, unless otherwise indicated.

Procedures

For all of the examples below, the paper used for sizing was prepared in advance, stored, and then treated on a laboratory puddle size press with the materials described. In all cases the base paper had no treatment applied at the size press during its manufacture. The application of materials at the size press consisted of dissolving starch in water by stirring and heating to about 95°C for at least 30 minutes. The starch solution was then kept at 65°C until used, usually within a few hours. In some cases, sodium chloride (up to about 0.7 wt.%) was added. Sodium chloride is a typical additive in paper mill size presses, where it is used to increase paper conductivity and thus reduce static charge build-up. The starch solution pH was adjusted to about pH 8 before use, and then the size press additives were added to the starch. In some cases, as noted below, the pH was readjusted at this point. The materials were mixed for a few minutes and then added to the nip of two rollers on the puddle size press.

The untreated paper was fed through the rollers one time to apply the solution in the nip to the paper. The amount of solution applied to the paper by a specific starch solution under specific conditions was determined and used to set the level of additives in the starch solution to give the desired level of paper treatment.

Immediately following the application of the size press composition, the papers were dried on a drum dryer heated at 93-105°C. The papers were then conditioned and tested.

Hercules Size Test: The Hercules Size Test, an art-recognized test for measuring sizing performance, is described in *Pulp and Paper Chemistry and Chemical Technology*, J.P. Casey, Ed., Vol. 3, p. 1553-1554 (1981) and in TAPPI Standard T530. The Hercules Size Test determines the degree of sizing by measuring the change in reflectance of the paper's surface as an aqueous solution of dye penetrates from the opposite surface side. The aqueous dye solution, *e.g.*, naphthol green dye in 1% formic acid, is contained in a ring on the top surface of the paper, and the change in reflectance is measured photoelectrically from the bottom surface.

Test duration is limited by choosing a convenient end point, *e.g.*, a reduction in reflected light of 20%, corresponding to 80% reflectance. A timer measures the time (in seconds) for the end point of the test to be reached. Longer times correlate with increased sizing performance, *i.e.*, resistance to water penetration increases.

Cobb Sizing Test: The Cobb sizing test is described in TAPPI standard T441. It consists of exposing a surface of the sample to water for a specified time and then measuring the amount of water absorbed by the increase in weight of the sample. Results are reported in grams of water absorbed per square meter per minute. Lower results correspond to higher levels of sizing.

Ink Jet Printing Evaluation: Ink jet printing was tested with a Hewlett Packard Deskjet 560C printer. A Hewlett Packard 3.4 test pattern and method were used to rate the quality of the printing.

Before testing the paper was conditioned at 23° C and 50% relative humidity for a minimum of one (1) day.

A. Evaluation of Black Ink Print Quality

Optical Density - An optical densitometer was placed over the black test rectangle on the printed sheet, and the optical density for black was recorded. This was repeated on different areas of the rectangle for a total of 6 readings.

Black Ink Spread (Feathering) - Black ink spread is the tendency for the ink to spread out from the print area. Using the magnifier, areas of the test pattern consisting of rows of the letter "E" were examined and the print quality was compared with standard examples of acceptable, good and unacceptable feathering. Specific areas that were examined were: degree of rounding of the square ends of the letter; amount of separation between the center stroke and the right ends of the letter, general breadth of the lines, etc. Similar inspection of line growth was made using the vertical and horizontal black lines in the test pattern.

Black Edge Roughness (Wicking) - Black edge roughness or wicking is the tendency for the ink to bleed away from the print area along a fiber or one direction, causing rough edges, even long "spidery" lines on the periphery of the print area. Using the magnifier, all areas of the test pattern where black lines are printed against a white background were examined and compared with the standard examples of acceptable, good and unacceptable wicking.

B. Evaluation of Color Print Quality

Optical Density -The optical densitometer was positioned over the composite black rectangle on the printed sheet, and the black optical density number was recorded. The composite black print consisted of a combination of cyan, magenta and yellow inks. The procedure was repeated on different areas of the rectangle for a total of 6 readings which were averaged and reported as composite black optical density.

Color -Color Edge Roughness - Color-color edge roughness measures the roughness of lines in areas where two colors overlap. Areas of the test pattern where composite black and yellow areas overlap were examined with a magnifier and compared with standard examples to judge whether the print quality was acceptable, good or unacceptable.

Color-Color Line Growth - Color-color line growth examines the size of printed features of one color touching or overlapping another color versus the intended size. A magnifier was used to examine the overlapping color text areas of the test pattern and to compare them with standard examples as acceptable, good or non-acceptable. Specifically, the size of composite black characters on a yellow background and yellow characters on a black background were examined.

Toner Adhesion: Relative toner adhesion is the relative amount of white paper showing through a solid black area of toner, applied by a copy machine, that results from the paper being creased. For the test, the paper was creased in a controlled fashion (toner on the inside of the crease), was unfolded, and then the loose toner was removed in a reproducible manner. The percentage of the crack area from which toner was lost was estimated by microscopic or optical density measurement of the crack and surrounding areas of toner, and reported as the toner adhesion value. Thus, a smaller value means that less toner is lost thus indicating greater toner adhesion.

Example 1

This example shows the results of surface sizing with a combination of Precis®2000 sizing agent, an aqueous dispersion of a ketene dimer liquid at 25°C, available from Hercules Incorporated, Wilmington, DE, and an aqueous dispersion of a pentaerythritol ester of rosin, Pentalyn®H synthetic resin, available from Hercules Incorporated, Wilmington, DE. Papers containing only Pentalyn®H and only Precis®2000 were also prepared for comparison.

The aqueous dispersion of Pentalyn®H was prepared by melting Pentalyn® H in a vessel fitted with means for heating and stirring and then heating it to a temperature of 185-195°C. An aqueous solution of cationic waxy maize starch (Sta-lok®J140, Staley Manufacturing Co., Decatur, IL) and sodium lignosulfonate, was prepared, which on mixing with Pentalyn H gave a size dispersion having a dry basis content of Pentalyn H, starch and sodium lignosulfonate in the ratio of 100 to 12.5 to 6.25 parts. The aqueous solution of the starch and lignosulfonate was prepared at a total solids content of 7-8%, preheated under pressure to 145-160°C and mixed with the resin in a pressurized system to give a dispersion with a total solids content of 34-36%. The product prepared in the first stage mixing process was refined, to reduce the droplet diameter to 1-2 microns, in a valve homogenizer, of the Manton Gaulin type.

The starch solution prepared contained 4% starch (D150 from Grain Processing Corp., Muscatine, IA), 0.3% sodium chloride. The levels of Pentalyn®H and Precis®2000 on a solids basis were adjusted such that the levels when applied to paper were as noted below. The pH of the final mixture was adjusted to between 7.5 and 8. It was applied to paper by the procedures described above.

The paper was made at a basis weight of 75 g/m^2 from pulp consisting of 75% hardwood and 25% softwood refined to 425 CSF. It contained 10% calcium carbonate filler, 0.5% Sta-Lok®400 cationic starch (available from Staley Manufacturing Co., Decatur, IL) and 0.25% alum all of which were added internally during the preparation of the paper.

When the Pentalyn®H was present as a surface size at the 0.25% level with no Precis®2000, the HST sizing was <1 sec. When the Pentalyn®H was present at the 0.25% level together with 0.012% Precis®2000, the HST sizing was 4 sec, thus demonstrating an improvement in sizing. Precis®2000 alone at the 0.012% level yielded paper with sizing of <1 sec in the HST test.

Example 2

This example shows the results of surface sizing with a combination of Precis®8023 sizing agent (available from Hercules Limited, Reigate, Surrey, UK), an aqueous dispersion of a ketene dimer liquid at 25°C and an aqueous dispersion of a pentaerythritol ester of rosin, Pentalyn®H synthetic resin, available from Hercules Incorporated, Wilmington, DE. Also included are data on ink jet printing performance and printing optical density.

The paper was made at a basis weight of 80 g/m^2. It contained calcium carbonate filler and internally added alkylketene dimer size.

An aqueous dispersion of Pentalyn®H was prepared as described in Example 1. For the surface sizing experiments the Pentalyn®H dispersion was blended with an aqueous emulsion of liquid ketene dimer, Precis®8023 sizing agent available from Hercules Incorporated, Wilmington, Delaware. The levels of the two dispersions used were such as to give proportions by weight on a dry basis of Pentalyn®H to liquid ketene dimer as shown in Table 1.

Table 1

Size Compositions

Sizing Agent	Rosin Ester (parts by weight)	Liquid Alkylketene Dimer (parts by weight)
rosin ester	100	0
liquid alkylketene dimer	0	100
A	80	20
B	60	40

The blended dispersions were mixed into an 8% solids solution of Perfectamyl®4692A starch (from Avebe b.a., Veendam, The Netherlands). The resulting size press starch solution containing the dispersions were warmed to 60°C and applied with a bench model puddle size press to both sides of the sheet. The paper ran through the press nip at 12 m/minute, and was dried on a drum drier at 105°C with a dwell time of 20 sec.

Tables 2 and 3 below show the properties of the paper for sizing and printing. Print properties were measured by image analysis of print quality on paper printed with the Hewlett Packard standard format with a Hewlett Packard HP Deskjet 560C printer. Image analysis was performed with Kontron KS 400 Image Analysis Software on a computer connected to a

Zeiss Stereo microscope Stemi 2000-C, equipped with a video camera. Optical densities were measured with a Gretag 182 Densitometer.

Table 2

Sizing, Ink Jet Performance and Optical Density

Surface Size	Addition Level (% dry basis)	Sizing HST, sec.	Color-to-Color Bleed			Optical Density	
			R_t	R_b	W	mono	composite
None		113	2.05	1.98	353	1.17	0.81
Starch only	2	144	1.91	1.91	315	1.33	0.94
Rosin ester	0.025	155	1.96	1.88	323	1.30	0.93
Rosin ester	0.05	180	2.1	1.93	325	1.30	0.92
A	0.025	182	2.08	2.02	323	1.35	0.94
A	0.05	191	1.99	2.04	316	1.30	0.93
B	0.025	188	2.02	1.99	320	1.33	0.89
B	0.05	183	2.14	1.95	324	1.29	0.88
Liquid ketene dimer	0.025	172	2.06	1.94	324	1.30	0.93
Liquid ketene dimer	0.05	155	2.08	2.05	321	1.33	0.93

Notes:

Ratios are measured at the top (R_t) and bottom (R_b) of the printed areas, the top being the position of the letter or line in relation to the printing head. W, the width reported, is the band width of the printed area measured in pixels. Higher values indicated more spreading and thus more diffuse printing quality. Higher values of R_t, R_b and W indicte a greater lateral spread of of ink as shown by the ratio of real length of the boundry and a straight line drawn across the same boundry.

Optical density of the print was measured witha Gretag 182 Densitometer. Higher values indicate improved quality. "Mono" is the print produced by the cartridge black pigment. "Composite" is the black color produced by the combination of all the pigments in the inkjet head of the Hewlett Packard printer.

Table 3
Black and White Feathering and Bleed Through

Surface Size	Addition Level (% dry basis)	Black and White Feathering			Bleed-Through (OD)	
		R_a	R_b	W	mono	composite
None		1.38	1.40	328	0.11	0.49
Starch only	2	1.33	1.36	329	0.1	0.39
Rosin ester	0.025	1.31	1.45	327	0.11	0.49
Rosin ester	0.05	1.45	1.36	323	0.11	0.55
A	0.025	1.50	1.37	323	0.1	0.46
A	0.05	1.46	1.36	324	0.11	0.43
B	0.025	1.48	1.37	318	0.10	0.46
B	0.05	1.52	1.36	323	0.10	0.54
Liquid ketene dimer	0.025	1.25	1.47	321	0.1	0.55
Liquid ketene dimer	0.05	1.37	1.53	321	0.11	0.51

Notes:
The feathering of black pigment on paper was measured by image analysis by the same method described for color-to-color bleed.
Bleed-through is measured by optical density on the reverse side of the paper. The optimum result is 0.

The results in evaluation Table 2 show that sizing with liquid ketene dimer and rosin ester is higher than that obtained when either of the components is used alone at the same addition level.

With regard to the ink jet printing results, at the 0.025% addtion level the mixed rosin ester and liquid ketene dimer gives somewhat higher black pigment ("mono") print optical density than do the components used alone.

Example 3

This example illustrates surface sizing with a combination of an aqueous dispersion of a ketene dimer liquid at 25°C (Precis®8023 sizing agent), and an aqueous dispersion of rosin. The rosin was provided in the form of Hi-Phase®35J, a high free rosin dispersion size available from Hercules Limited, Reigate, Surrey, UK.

To prepare the size mixture 150.65 parts of Hi-Phase®35J, high free rosin dispersion size was charged to a stirred reaction vessel, and then 100 parts of Precis®8023 sizing agent was added. The ratio of rosin to liquid ketene dimer in the resulting size mixture was 2:1.

Paper used for the sizing tests was made at a basis weight of 65 g/m^2 with carbonate filler and no internal size. For the sizing tests the appropriate size (either the size mixture or Precis®8023 alone) was mixed into a starch size press solution and applied onto the surface of the paper to give a pick up weight of 0.6% on a dry basis. The size press solution was warmed to 60°C and applied with a bench model puddle size press to both sides of the sheet. The paper ran through the press nip at 12 m/min and was dried on a drum drier at 105°C with a dwell time of 20 seconds. The size press starch solution of Perfectamyl 4692A starch was adjusted to 8% total solids after cooking.

Tables 4 below shows the sizing and printing properties of the paper. Sizing was measured by the Cobb method (Tappi method T441OM/90 Revised 1990). In the Cobb test lower values correspond to higher sizing. Print properties were measured by image analysis of print quality on paper printed with the standard format with a Hewlett Packard HP Deskjet 560C. Image analysis was performed with Kontron KS 400 Image Analysis software run on a computer connected to a Zeiss Stereo microscope, Stermi 2000-C, equipped with a video camera. Optical densities were measure with a Gretag 182 densitomer.

Table 4

Size	Cobb Sizing (g/m^2/min.)	Color-to Color Bleed			Optical Density
		R	R	W	composite
Size Mixture	23.5	1.74	1.91	282	0.94
Precis®8023	24.4	2.11	2.07	278	1.12

These results show that the blend of liquid ketene dimer with rosin is a more efficient sizing material than the same weight of liquid ketene dimer. The blend gave better sizing, better ink jet color print quality and held out the print colors against bleed-through more effectively than the same weight of liquid ketene dimer.

It is not intended that the examples presented here should be construed to limit the invention, but rather they are submitted to illustrate some of the specific embodiments of the invention. Various modifications and variations of the present invention can be made without departing from the scope of the appended claims.

What is claimed is:

1. A process for preparing sized paper comprising:

a) providing an aqueous pulp suspension;

b) sheeting and drying the aqueous pulp suspension to obtain paper;

c) applying to the paper a size composition comprising at least one 2-oxetanone that is not solid at 25°C and at least one rosin; and

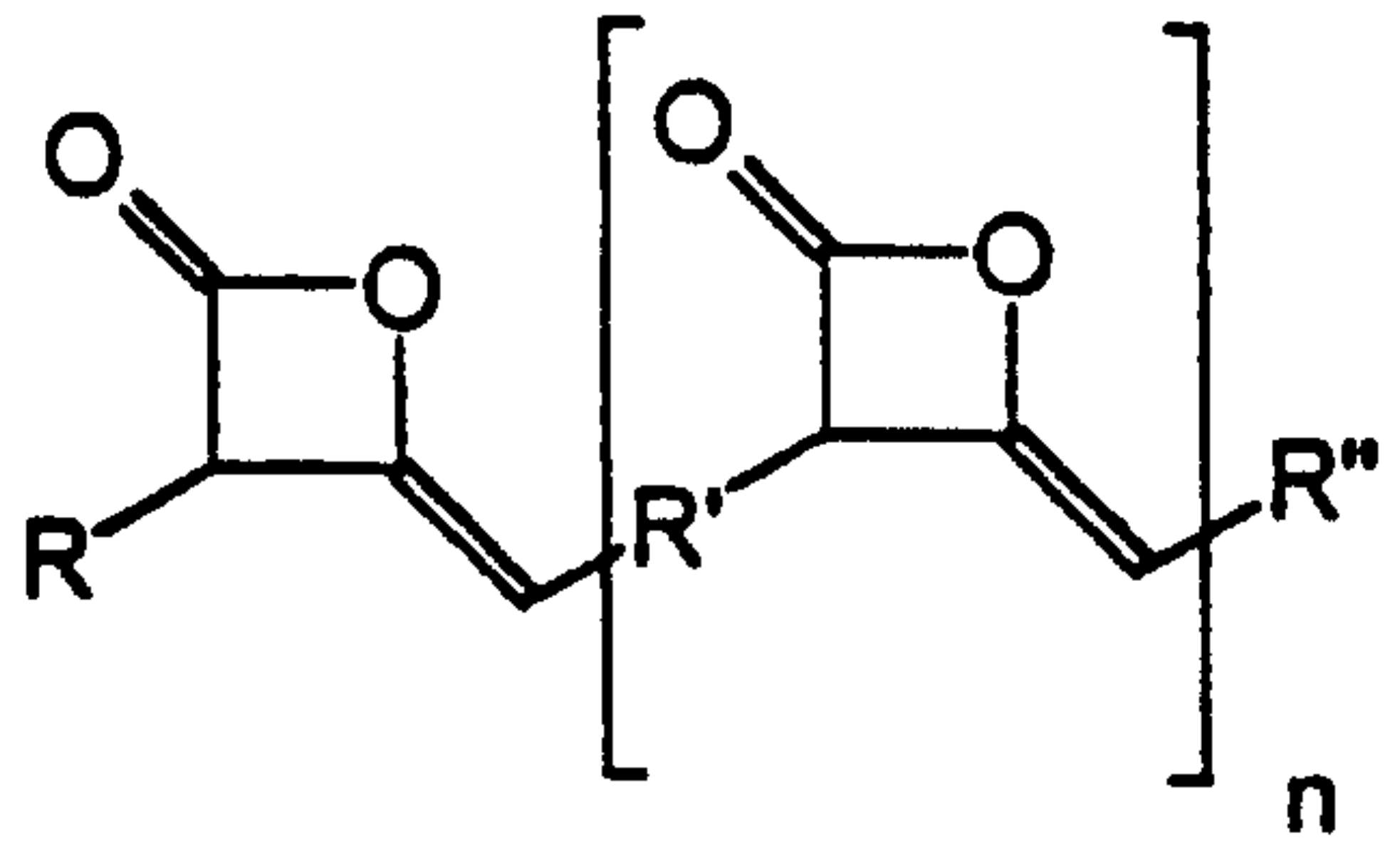
d) drying the paper.

2. The process of claim 1 wherein the 2-oxetanone is not solid at 20°C.

3. The process of claim 1 wherein the 2-oxetanone is liquid at 25°C.

4. The process of claim 1 wherein the 2-oxetanone is liquid at 20°C.

5. The process of claim 1 wherein the 2-oxetanone comprises a mixture of compounds having the structure:



wherein n is an integer of 0 to about 20, R and R", which may be the same or different, are saturated or unsaturated straight chain or branched alkyl groups having 6 to 24 carbon atoms; and R' is a saturated or unsaturated straight chain or branched alkyl group having from about 2 to about 40 carbon atoms, and wherein at least 25% of the R and R" groups is unsaturated.

6. The process of claim 5 wherein R and R" have from 10 to 20 carbon atoms and R' has from 4 to 8 or from 28 to 40 carbon atoms.

7. The process of claim 5 wherein R and R" have from 14 to 16 carbon atoms and R' has from 4 to 8 or from 28 to 40 carbon atoms.

8. The process of claim 5 wherein n is an integer of from 0 to about 6.

9. The process of claim 5 wherein n is an integer of from 0 to about 3.

10. The process of claim 1 wherein the rosin is selected from the group consisting of unmodified rosin, fortified rosin, rosin ester, hydrogenated rosin and extended rosin.

11. The process of claim 1 wherein the rosin is an ester of rosin and an alcohol selected from the group consisting of glycerol, ethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, 1,4-butanediol, sorbitol and mannitol.

12. The process of claim 1 wherein the rosin is an ester of rosin and an alcohol selected from the group consisting of glycerol and pentaerythritol.

13. The process of claim 1 wherein the rosin is an ester of rosin and pentaerythritol.

14. The process of claim 1 wherein the ratio on a dry basis of rosin to 2-oxetanone is from about 0.2:1 to about 50:1.

15. The process of claim 1 wherein the ratio on a dry basis of rosin to 2-oxetanone is from about 0.5:1 to about 40:1.

16. The process of claim 1 wherein the ratio on a dry basis of rosin to 2-oxetanone is from about 1:1 to about 30:1.

17. The process of claim 1 wherein the size composition is a dispersion in water of rosin and 2-oxetanone that is not solid at 25°C.

18. The process of claim 17 wherein the dispersion is prepared by mixing aqueous dispersions of rosin and 2-oxetanone.

19. The process of claim 1 wherein the size composition further comprises at least one salt of a cationic metal ion that is soluble in water at a pH of about 7 to about 9.

20. The process of claim 19 wherein the at least one water-soluble salt is selected from the group consisting of sodium chloride, sodium sulfate, calcium chloride, calcium bromide, magnesium chloride, magnesium bromide, aluminum sulfate and poly aluminum chloride.

21. The process of claim 19 wherein the weight ratio of the at least one water-soluble salt to the other non-aqueous components of the size composition is from about 1:20 to about 20:1.

22. The process of claim 19 wherein the weight ratio of the at least one water-soluble salt to the other non-aqueous components of the size composition is from about 1:5 to about 5:1.

23. The process of claim 19 wherein the weight ratio of the at least one water-soluble salt to the other non-aqueous components of the size composition is from about 1:3 to about 3:1.

24. The process of claim 1 wherein the size composition further comprises starch.

25. The process of claim 24 wherein the starch in the size composition is at a level of about 1 wt.% to about 20 wt.% on a dry basis based on the total weight of the aqueous size composition.

26. The process of claim 24 wherein the starch in the size composition is at a level of about 2 wt.% to about 16 wt.% on a dry basis based on the total weight of the aqueous size composition.

27. The process of claim 24 wherein the starch in the size composition is at a level of about 3 wt.% to about 12 wt.% on a dry basis based on the total weight of the aqueous size composition.

28. The process of claim 1 wherein the total of rosin and 2-oxetanone in the size composition is at a level of from about 0.01 to about 2 wt.% on a dry basis based on the total weight of the aqueous size composition.

29. The process of claim 1 wherein the total of rosin and 2-oxetanone in the size composition is at a level of from about 0.02 to about 0.1 wt.% on a dry basis based on the total weight of the aqueous size composition.

30. The process of claim 1 wherein the size composition is applied at a level that provides from about 0.01 wt.% to about 1 wt.% total rosin and 2-oxetanone on a dry basis, based on the weight of the dry paper.

31. The process of claim 1 wherein the size composition is applied at a level that provides from about 0.03 wt.% to about 0.7 wt.% total rosin and 2-oxetanone on a dry basis, based on the weight of the dry paper.

32. The process of claim 1 wherein the size composition is applied at a level that provides about 0.05 wt.% to about 0.5 wt.% total rosin and 2-oxetanone on a dry basis, based on the weight of the dry paper.

33. The process of claim 24 wherein the size composition is applied at a level that provides about 1 wt.% to about 8 wt.% starch on a dry basis based on the dry weight of the paper.

34. The process of claim 24 wherein the size composition is applied at a level that provides about 2 wt.% to about 7 wt.% starch on a dry basis based on the dry weight of the paper.

35. The process of claim 24 wherein the size composition is applied at a level that provides about 3 wt.% to about 6 wt.% starch on a dry basis based on the dry weight of the paper.

36. The process of claim 1 wherein the size composition is applied at a level such that the level of the 2-oxetanone applied is from about 0.005 to about 0.5 wt.% on a dry basis based on the weight of the dry paper.

37. The process of claim 1 wherein the size composition is applied at a level such that the level of the 2-oxetanone applied is from about 0.01 to about 0.3 wt.% on a dry basis based on the weight of the dry paper.

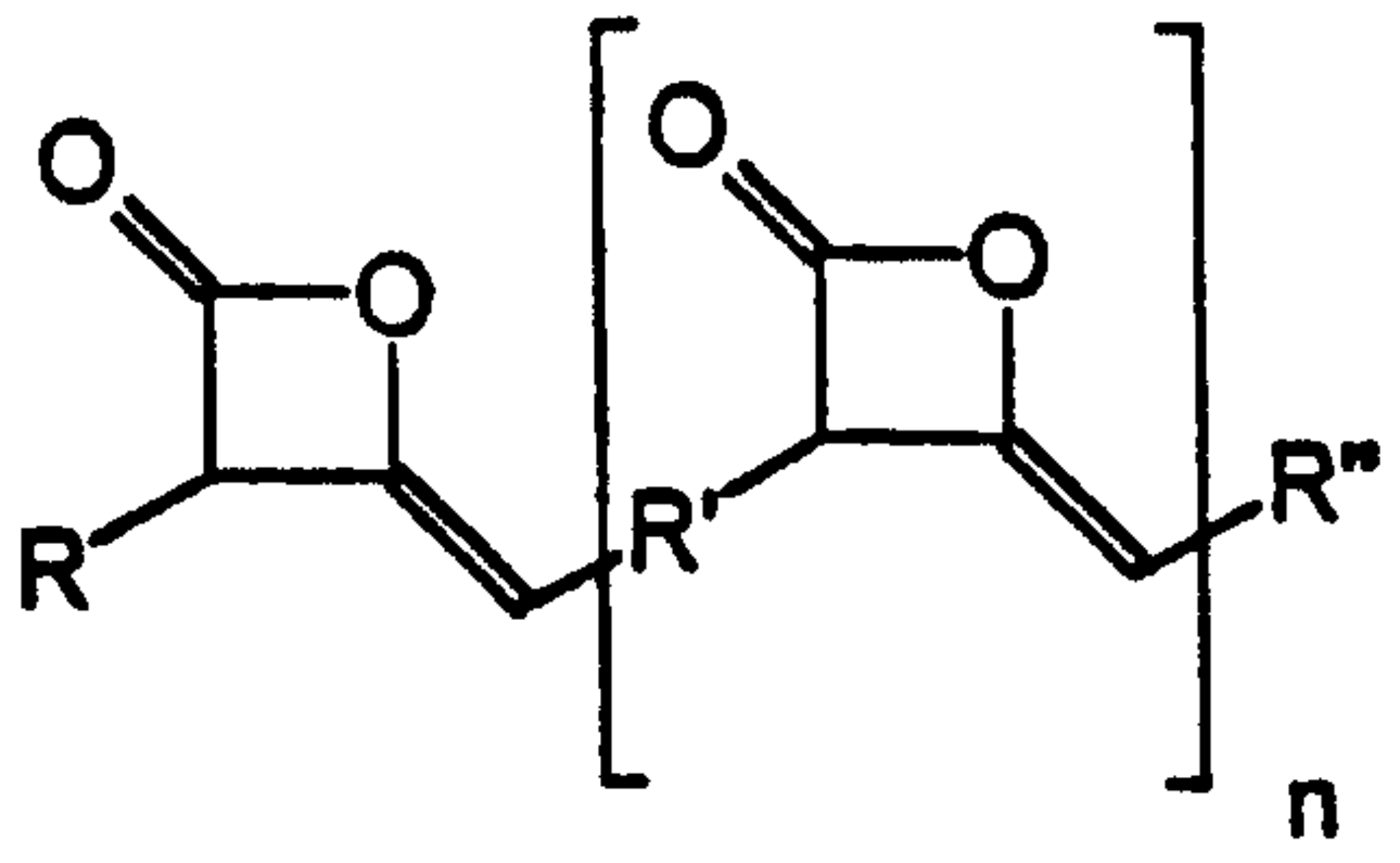
38. The process of claim 1 wherein the size composition is applied at a level such that the level of the 2-oxetanone applied is from about 0.02 to about 0.2 wt.% on a dry basis based on the weight of the dry paper.

39. The process of claim 1 wherein the size composition is applied at a level such that the level of the rosin applied is from about 0.01 to about 0.5 wt.% on a dry basis based on the weight of the dry paper.

40. The process of claim 1 wherein the size composition is applied at a level such that the level of the rosin applied is from about 0.02 to about 0.4 wt.% on a dry basis based on the weight of the dry paper.

41. The process of claim 1 wherein the size composition is applied at a level such that the level of the rosin applied is from about 0.04 to about 0.3 wt.% on a dry basis based on the weight of the dry paper.

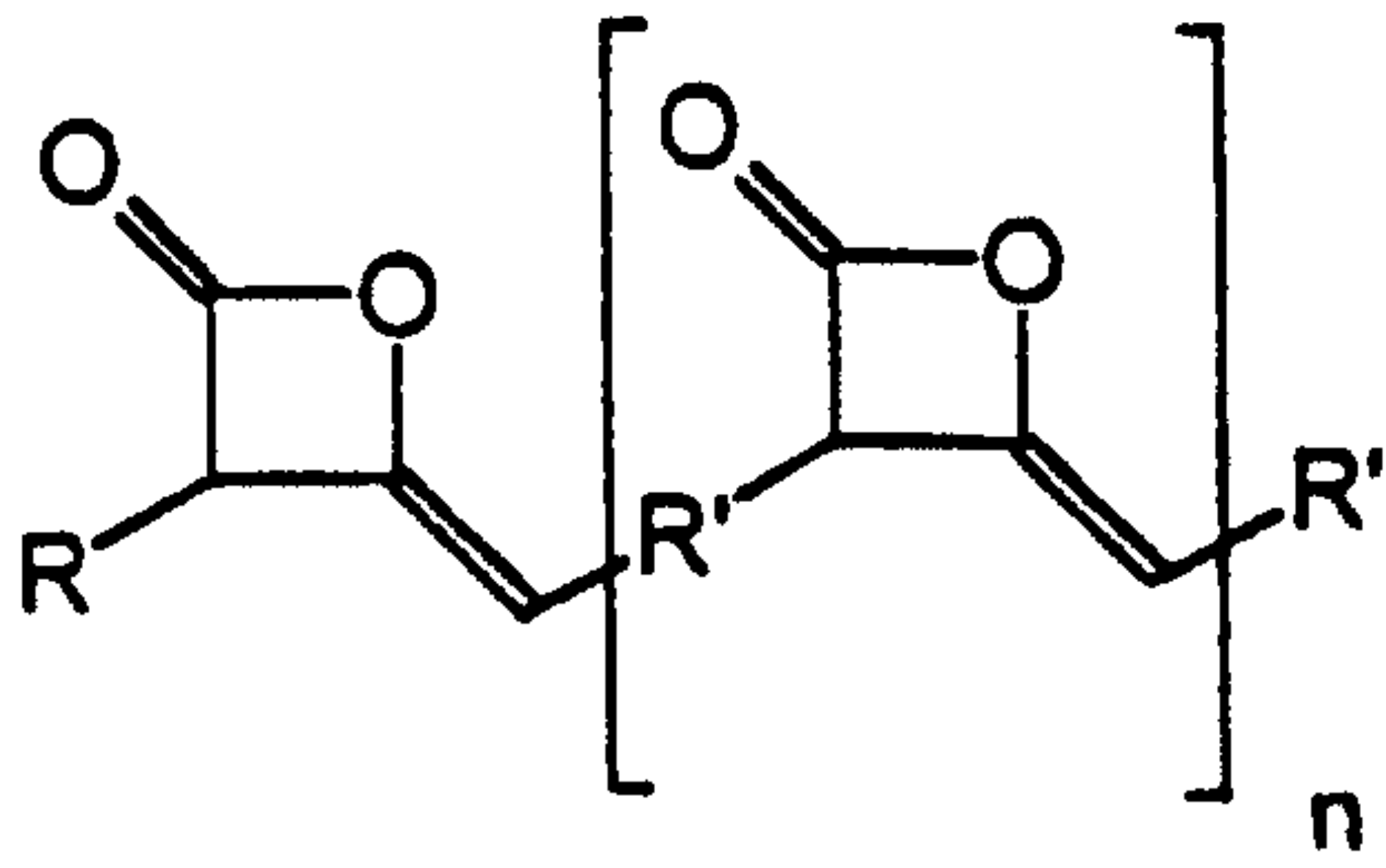
42. The process of claim 1 wherein the 2-oxetanone comprises a mixture of compounds having the structure:



wherein n is an integer of 0 to about 20, R and R", which may be the same or different, are saturated or unsaturated straight chain or branched alkyl groups having 6 to 24 carbon atoms; and R' is a saturated or unsaturated straight chain or branched alkyl group having from about 2 to about 40 carbon atoms, and wherein at least 25% of the R and R" groups is unsaturated; and

the rosin is selected from the group consisting of unmodified rosin, fortified rosin, rosin ester, hydrogenated rosin and extended rosin.

43. The process of claim 1 wherein the 2-oxetanone comprises a mixture of compounds having the structure:



wherein n is an integer of 0 to about 20, R and R", which may be the same or different, are saturated or unsaturated straight chain or branched alkyl groups having 6 to 24 carbon atoms; and R' is a saturated or unsaturated straight chain or branched alkyl group having from about 2 to about 40 carbon atoms, and wherein at least 25% of the R and R" groups is unsaturated;

the rosin is selected from the group consisting of unmodified rosin, fortified rosin, rosin ester, hydrogenated rosin and extended rosin;

the ratio on a dry basis of rosin to 2-oxetanone in the size composition is from about 0.2:1 to about 50:1;

the total of rosin and 2-oxetanone in the size composition is at a level of from about 0.01 to about 2 wt.% on a dry basis based on the total weight of the aqueous size composition; and

the size composition is applied at a level that provides from about 0.01 wt.% to about 1 wt.% total rosin and 2-oxetanone on a dry basis, based on the weight of the dry paper.

44. The process of claim 1 wherein the applying of step (c) takes place at a size press.

45. The process of claim 44 wherein the size press is a puddle size press.

46. The process of claim 44 wherein the size press is a gate roller size press.

47. The process of claim 44 wherein the size press is a metered blade size press.

48. The process of claim 1 further comprising adding at least one size to the aqueous pulp suspension prior to step (b).

49. The process of claim 48 wherein the at least one size is selected from the group consisting of rosin size, 2-oxetanones and alkenylsuccinic anhydrides.

50. Sized paper made by the process of claim 1.

51. The sized paper of claim 50 that exhibits better water holdout than paper that is the same except that the sizing composition contains only rosin.

52. The sized paper of claim 50 that performs better in ink jet printing than does paper that is the same except that the aqueous size composition contains only 2-oxetanone or only rosin when the printing is evaluated for at least one property selected from the group consisting of optical density, feathering, wicking, edge roughness and bleed.

53. The sized paper of claim 50 that has higher toner adhesion than does paper that is the same except that the size composition contains only 2-oxetanone.

54. The sized paper of claim 50 that has a higher static or kinetic coefficient of friction than does paper that is the same except that the size composition contains only 2-oxetanone.

55. The sized paper of claim 50 that has a lower COF bandwidth than does paper that is the same except that the size composition contains only 2-oxetanone.