

[54] **BETA-DISULFONE SILVER HALIDE SOLUBILIZING AGENTS**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 42,606, June 1, 1970, abandoned.

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[51] Int. Cl. **G03c 5/38**

[58] Field of Search **96/61, 29**

[56]

References Cited

UNITED STATES PATENTS

3,220,839	11/1965	Herz	96/61
3,241,966	3/1966	Heilmann.....	96/61 X
3,169,992	2/1965	Henn.....	96/61 R

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[57]

ABSTRACT

β -disulfones are employed as silver halide solubilizing agents for use in fixing compositions, and as silver halide solvents in photographic chemical diffusion transfer processes, photosensitive elements, photographic film units and reception elements.

14 Claims, No Drawings

BETA-DISULFONE SILVER HALIDE SOLUBILIZING AGENTS

This is a continuation-in-part application of U.S. application, Ser. No. 42,606 of Paul H. Stewart and Donald W. Heseltine, filed June 1, 1970, now abandoned.

This invention relates to the art of photography. More particularly, this invention relates to novel silver halide solubilizing agents.

In photographic processes, after the development of exposed photographic silver halide emulsion layers, the developed photograph contains residual silver halide that has not been affected by the developer. In order to obtain a relatively permanent image, the silver halide is converted by fixation into a soluble compound which can be removed by washing. If the residual silver halide were left in the developed emulsion layer, it would be converted to silver upon exposure to light and thereby obliterate the image. Accordingly, it is common to fix a developed photographic silver halide emulsion layer by employing a fixing agent which forms a water-soluble complex which can be washed out of the emulsion layer along with the fixing agent. Thus, for example, thiouslfate in the form of a sodium or ammonium salt is the most commonly used fixing agent. Other fixing agents include thiocyanates, certain mercaptans, e.g., mercapto acetic acid, and complexes with divalent metals, such as the complex formed by zinc acetate, and diethylaminoethane thiol have been employed as fixing agents. Fixing agents constitute one class of silver halide solubilizing materials.

Silver halide solubilizing materials are also used in chemical transfer processes wherein unexposed and undeveloped silver halide is transferred to a receiving sheet or a web. The solubilizing agent facilitates the transfer of the silver halide to the web. Such transfer processes are well known in the art and are exemplified by the Rott U.S. Pat. No. 2,352,014; and the Land U.S. Pats. Nos. 2,543,181; 2,584,029; and 2,698,236. Likewise, silver halide solubilizing agents have been employed in monobaths wherein a single processing composition can be utilized to both develop and fix a photographic silver halide emulsion layer. Such compositions contain a silver halide developing agent in addition to the silver halide solubilizing agent.

It is an object of this invention to provide novel compositions having high silver halide solvent activity.

It is another object of this invention to provide a novel process for fixing developed photographic silver halide emulsion layers.

It is still another object of this invention to provide a new photographic chemical transfer process wherein a novel silver halide solvent is utilized.

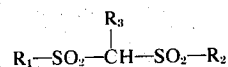
Still another object of the invention is to provide photosensitive elements photographic film units and reception elements having a novel silver halide solvent incorporated therein.

These and other objects of the invention are accomplished by employing 1,1 bis-sulfonyl alkanes (or β -disulfones) as silver halide solvents in various applications described herein. It has been found that the β -disulfones used in the present invention are excellent fixing agents and can be employed in any photographic process or product wherein silver halide solvents are ordinarily employed.

The β -disulfones and their chemical reactions have been described in the literature. For example, the preparation of these compounds and reactions in which

they are involved are described in Suter, *Organic Chemistry of Sulfur*, on pages 735 and 739. However, it has now been discovered that particular β -disulfones, e.g., 1,1 bis-sulfonyl alkanes, are excellent silver halide solvents.

Suitable 1,1 bis-sulfonyl alkanes used in the present invention can be represented by the general formula



wherein R_1 and R_2 each represent a lower alkyl group and R_3 represents H or a lower alkyl group. The term "lower alkyl" as employed herein is preferably an alkyl group containing from about one to four carbon atoms, e.g., methyl, ethyl, propyl, isopropyl, butyl and the like. The term "alkyl" as employed herein also includes substituted alkyl groups, and is preferably a substituted lower alkyl group containing from one to four carbon atoms. Thus, substituted lower alkyl groups such as a hydroxyalkyl group, e.g., β -hydroxyethyl, ω -hydroxybutyl, etc.; an alkoxyalkyl group, e.g., β -methoxyethyl, ω -butoxybutyl, etc.; a carboxyalkyl group, e.g., β -carboxyethyl, ω -carboxybutyl, etc.; a sulfoalkyl group, e.g., β -sulfoethyl, ω -sulfobutyl, etc.; a sulfatoalkyl group, e.g., β -sulfatoethyl, ω -sulfatobutyl, etc.; an acryloxyalkyl group, e.g., β -acetoxyethyl, ω -butyryloxybutyl, etc; alkoxy-carbonylalkyl group, e.g., β -methoxycarbonyl-ethyl, ω -ethoxy-carbonylbutyl, etc. are preferred.

1,1 Bis-sulfonyl alkanes within the above formula include 1,1 bis-(methylsulfonyl)ethane; 1,1 bis-(ethylsulfonyl)-ethane; 1,1 bis-(methylsulfonyl)propane; 1,1 bis-(ethylsulfonyl)-propane; 1-ethylsulfonyl-1-methylsulfonyl methane; 1-ethyl-sulfonyl-1-methylsulfonyl ethane; etc. Especially preferred 1,1 bis-sulfonyl alkanes are bis(methylsulfonyl)methane, bis(ethyl-sulfonyl)methane, bis(β -hydroxyethylsulfonyl)methane, bis(carboxymethylsulfonyl)methane, and bis(β -carboxyethylsulfonyl)-methane.

The subject silver halide solubilizing agents can be utilized in aqueous compositions containing at least about one gram of disulfone per liter of composition. The amount of such silver halide solubilizing agent can be widely varied, the amount used being readily ascertainable by one skilled in the art for the particular photographic application. Generally, however, the amount of disulfone utilized will vary between about 3 grams and about 200 grams per liter of processing composition.

When the subject disulfones are employed in baths for clearing or fixing developed photographic film, at least 5 or 10 to 200 grams of the disulfone per liter of processing solution are more generally utilized to obtain fixing with a practical time interval. Lower concentrations, e.g., down to about 3 grams per liter, are more generally utilized when the subject disulfone is employed as a silver halide solvent in a chemical transfer process.

The subject disulfones exhibit fixing or clearing activity in alkaline or basic compositions. Generally, alkaline solutions having pH's of at least about 8 or 9, preferably at least 11, are employed. However, the alkalinity of the processing compositions can be varied over a wide range in accordance with usual practice. Alkaline materials, such as alkaline metal carbonates and

hydroxides and amines can be utilized to attain the desired alkalinity.

A photosensitive element according to the invention comprises a support having thereon a silver halide emulsion layer, the element also containing the novel disulfones described herein either in the silver halide emulsion layer or in a separate layer. The disulfones can be present in any amount suitable for the intended purpose and can be varied over a wide range, i.e., the amount of disulfone present is dependent upon how much silver halide is present initially in the photosensitive element and how much of it is desired to be dissolved or solubilized. The ability to incorporate the novel disulfones described herein in a photosensitive element is an important feature of the invention. Prior art silver halide solvents, such as hypo, cannot be successfully incorporated in a photosensitive element, especially in the silver halide emulsion layer, since it dissolves silver halide upon contact. The novel disulfones described herein, however, are relatively inert at pH's below about 8 or 9 and can be coated at the pH or below. However, they become active silver halide solvents above the pH. Alkaline activation of the novel silver halide solvents described herein thus serves as a very effective control of solubilizing activity.

A photographic film unit according to the invention comprises:

- a. a photosensitive element comprising a support having thereon a silver halide emulsion layer,
- b. a reception element comprising a support having thereon a silver halide precipitating agent and adapted to be superposed on the photosensitive element after exposure thereof, and
- c. a rupturable container containing an alkaline processing composition and adapted to be positioned between the photosensitive element after exposure thereof and the reception element during processing, so that a compressive force applied to the container, such as by pressure-applying members which would be found in a camera, will effect a discharge of the container's contents between the photosensitive element and the reception element, the film unit containing a 1,1 bis-sulfonyl alkane silver halide solubilizing agent. The novel disulfones can be located anywhere in the film unit, i.e., in the silver halide emulsion layer or in a layer contiguous thereto, in the receiving element or in the processing composition. The concentration of the disulfone present can be widely varied, as explained above, depending upon its location in the system, the amount of silver halide to be dissolved, etc.

As previously mentioned, the subject disulfones can be desirably employed as silver halide solvents in photographic chemical transfer processes. A transfer process for preparing reversal images is described in U.S. Pat. No. 2,352,014 to Rott, the disclosure of which is hereby incorporated by reference. In a chemical transfer process according to the invention, an exposed photographic silver halide emulsion is impregnated with a developer and pressed in contact with a receiving sheet in the presence of a silver halide solvent and a fogging agent. The undeveloped silver halide in the emulsion layer is transferred or diffuses by means of the silver halide solvent to the receiving sheet, this transferred silver halide developing to silver to form a positive image on the receiving sheet.

Other investigators in this field disclose the use of various specific fogging agents or nuclei that can be used in the reception layer to facilitate the formation of a silver image therein and the use of viscous processing materials to facilitate the utilization of processing materials in pods positioned between the light-sensitive negative layer and the reception layer.

A wide variety of nuclei or silver halide precipitating agents can be utilized in the reception layers used in silver halide solvent transfer processes. Such nuclei are incorporated into conventional photographic organic hydrophilic colloid layers such as gelatin and polyvinyl alcohol layers and include such physical nuclei or chemical precipitants as:

- a. heavy metals, especially in colloidal form and salts of these metals,
- b. salts, the anions of which form silver salts less soluble than the silver halide of the photographic emulsion to be processed, and
- c. nondiffusible polymeric materials with functional groups capable of combining with and insolubilizing silver ions.

Typical useful silver precipitating agents include sulfides, selenides, polysulfides, polyselenides, thiourea and its derivatives, mercaptans, stannous halides, silver, gold, platinum, palladium, mercury, colloidal silver, aminoguanidine sulfate, aminoguanidine carbonate, arsenous oxide, sodium stannite, substituted hydrazines, zanthates, and the like. Polyvinyl mercaptoacetate is an example of a suitable nondiffusing polymeric silver precipitant. Heavy metal sulfides such as lead, silver, zinc, aluminum, cadmium and bismuth sulfides are useful, particularly the sulfides of lead and zinc alone or in an admixture, or complex salts of these with thioacetamide, dithio-oxamide or dithio-biuret. The heavy metals and the noble metals particularly in colloidal form are especially effective. Other silver precipitating agents will occur to those skilled in the present art.

Such silver precipitating or nucleating agents can be utilized in reception layers that are separate elements from the light-sensitive silver halide negative element being processed, or they can be utilized as reception layers integral with the light-sensitive negative element. When the reception layer is utilized as a separate element, the reception layer is typically in the form of a web imbibed with processing material of the type suitable for processing long units of film such as motion picture film, or in the form of a support for receiving positive images of the "snap-shot" type that are processed in accordance with U.S. Pat. Nos. 2,543,181; 2,584,029 and 2,698,236. Belgian Patent 472,243 and U.S. Pat. No. 3,179,517 describe typical solvent transfer processes utilizing webs as a silver halide reception means. When the reception layer is integral with the light-sensitive negative element, such reception layer is generally a sublayer over which is coated the silver halide in a vehicle that is readily removable during processing such as a hydrophilic colloid soluble in aqueous alkali like cellulose ether phthalate.

Due to the inertness of the disulfones as described above, they can also be incorporated in a receiving element for chemical transfer processes. A receiving element according to the invention thus comprises a support having thereon a layer comprising physical development nuclei or silver precipitating agents, the element also containing the novel disulfones described

herein either in the nuclei layer or in a separate layer of the receiving element. Again, the concentration of the disulfone can be varied over a wide range depending upon the amount of silver halide present in the system to be dissolved.

Another patent disclosing photographic uses in which the subject disulfones can be employed is U.S. Pat. No. 3,220,838, the disclosure of which is also hereby incorporated by reference.

The subject disulfone silver halide solubilizing agents provide high speed fixing times, e.g., substantially complete fixation is obtained with relatively small amounts of disulfone within about 2 minutes.

The following examples further illustrate the present invention.

EXAMPLE 1-11

Several strips of a fine-grain, blue-sensitive, gelatino-silver chloride, developing-out, negative emulsion coated on a baryta paper support are treated at about 68°F. with alkaline compositions containing bis(methylsulfonyl)methane employing the various alkaline media set forth in Table I, below. The compositions contain 5.7 percent based on the anhydrous weight of each alkali compound, unless otherwise indicated. The bis(methylsulfonyl)methane is employed in a concentration of 172 milligrams/10 milliliters of the fixing composition.

TABLE I

Example No.	Alkaline Media	Approximate pH	Fixing Time (min)	Residual Silver Halide (mg./Ag/ft ²)
1	No treatment			96.0
2	H ₂ O (Control) ¹	6.0	90	96.0
3	H ₂ O ²	6.0	90	96.0
4	KOH	14.0	2	2.9
5	NaOH	14.0	2	2.2
6	NaOH(0.1N)	12.7	2	3.1
			6	1.2
7	NaOH(0.01N) ²	11.6	2	69.0
			6	50.0
8	Na ₂ PO ₄	12.3	2	0.4
9	Na ₂ CO ₃ ²	11.5	2	42.0
			6	24.0
10	Sodium metaborate	11.2	2	49.0
			6	33.0
11	NaOH(0.1M) ²	11.7	2	5.3
			4	3.1
			6	3.0

¹Na₂HPO₄*(0.1M)²

¹no bis(methylsulfonyl)methane present
²not all of the compound was dissolved

Following the fixing treatment, the samples are dipped into 3 percent acetic acid for a period of 15 minutes, following which the samples are washed with water for five minutes, air dried and then analyzed for silver. As seen by the results set forth in Table I, the fixing times or clearing times, i.e., the time interval necessary to dissolve the silver halide away from the photographic paper, is as low as 2 minutes when the bis(methylsulfonyl)methane fixing agent of the present invention is employed. On the other hand, when the disulfone fixing agent of the present invention is not employed, essentially no fixing or clearing of the photographic paper occurs.

EXAMPLE 12

This example illustrates the use of the disulfones of the present invention as silver halide solubilizing agents in producing a positive image in a photographic chemi-

cal transfer process. A fine-grain, negative-type, developing-out, gelatino-silver bromiodide emulsion layer on a cellulose acetate film support that had been exposed in an intensity scale sensitometer is processed employing the processing composition of Table II, below:

TABLE II

Ingredient	Concentration (grams)
N-methyl-p-aminophenol sulfate	1.5
Hydroquinone	2.5
Sodium sulfite, anhydrous	60.0
Trisodium phosphate	40.0
Bis(methylsulfonyl)methane	20.0
Water to make one liter	

The exposed film is immersed for about 30 seconds in the processing composition and is then pressed in contact with a receiving sheet comprising a cellulose acetate film support having coated thereon a gelatin layer containing nickel sulfide nuclei. After 60 seconds at a temperature of 70°F., the receiving sheet and the negative film are stripped apart and a positive dense brown-black silver image is found on the receiver.

EXAMPLE 13

This example illustrates the use of the present disulfone silver halide solubilizing agents in monobaths that are imbedded on processing webs used to develop and fix silver halide emulsion layers. A processing web is prepared by coating a layer comprising 2 grams of gelatin and 8 milligrams of Carey Lea silver per square foot on a cellulose acetate support. This web is soaked for about 5 minutes in a mono-bath processing composition having the ingredients indicated in Table III below:

TABLE III

Ingredient	Concentration (grams)
1-Phenyl-3-pyrazolidone	1.0
Hydroquinone	10.0
2-Dimethylaminoethanolsulfate (20 mol percent SO ₂)	174.0
Bis(methylsulfonyl)methane	10.0
Potassium bromide	2.0

Water to make one liter and pH adjusted to 11.5 with NaOH A high-speed coarse-grain, negative-type, developing-out, gelatino-silver bromiodide emulsion is coated onto a cellulose acetate film support and is exposed. Next, the film is processed by bringing the silver halide emulsion layer of the film in intimate contact with the nucleated layer of the soaked web and by allowing the resulting rolled sand-wich to be maintained for 10 minutes at 75°F. Thereafter the web and the film are peeled away to leave a fully developed and fixed negative film having a negative silver image in the exposed areas and being cleared of silver halide in the unexposed areas. A processing web which is spooled or sandwiched with a photographic film to be processed is conventional in the art and is more fully described in column 5 of U.S. Pat. No. 3,220,838 to Humphlett et al.

As shown in Example 13, the present silver halide solubilizing agents can be utilized in monobaths to both develop and fix an exposed photographic silver halide emulsion. These monobaths contain both a photographic developing agent, such as hydroquinone, that is suitable for developing or reducing latent silver halide images to silver, and a silver halide solubilizing or fixing agent (conventionally this has been, for example, sodium thiosulfate). As previously illustrated, the disul-

fone solubilizing agents of the present invention may be suitably employed in lieu of the conventional thiosulfate solubilizers and the like in such monobath compositions.

EXAMPLE 14

A developing composition is prepared having the following formula:

Bis(methylsulfonyl)methane	70.0 g.
Na ₂ SO ₃	20.0 g.
NaI	2.0 g.
NaOH	35.0 g.
Dihydroanhydro piperidino hexose reductone	20.0 g.
Hydroxyethyl cellulose (Natrasol 250H, sold by Hercules Powder Co., U.S.A.)	25.0 g.
H ₂ O to 1 liter	

Cysteine is then added to separate portions of this composition.

The developing compositions are used to process a photosensitive element comprising a coarse grain, gelatino silver bromiodide emulsion on a paper support which is exposed through a graduated density test tablet on an Eastman 1B sensitometer. The developing compositions are spread uniformly between the samples of the exposed photosensitive element and two separate receiving elements, as identified below, held in contact for 30 seconds and then separated. The receivers containing the transferred silver images are then cut in half and one-half is placed in a light stability chamber illuminated with fluorescent light at an intensity of 300 ft. candles, at 27°C., 90 percent relative humidity. After seven days in the chamber, characteristic curves of the silver images are compared to those of the images which are not subjected to the light stability chamber. The observations are recorded in the following table.

Receiver ¹	Control Developer (no cysteine)				Control Developer + 4.0 g./l. cysteine				Control Developer + 3.0 g./l. cysteine			
	Fresh D _{max}	7 Day D _{max}	Min. Info. Pt. ²	Den. Loss at 0.6 ³	Fresh D _{max}	7 Day D _{max}	Min. Info. Pt. ²	Den. Loss at 0.6 ³	Fresh D _{max}	7 Day D _{max}	Min. Info. Pt. ²	Den. Loss at 0.6 ³
I	1.58	1.22	0.28	0.34	1.20	1.12	0.19	0.14	1.12	1.06	0.16	0.24
II	1.52	1.16	0.16	0.23	1.20	1.15	0.08	0.14	1.18	1.08	0.22	0.24

¹ Receiver I is a polyethylene coated paper support on which is coated, in sequence, a cellulose triacetate layer, a gelatin sublayer, a polyacrylic acid layer, a cellulose acetate timing layer, a sublayer and a palladium nuclei receiving layer. Receiver II is like I but without the acid and timing layers.

² The minimum information point is that density value of the fresh image below which all information is lost on bleaching of the image after 7 days' keeping in the light stability chamber.

³ The loss in image density of the coating which is kept for 7 days in the light stability chamber which is measured at the 0.6 density point of the fresh coating.

The described β -disulfones can be employed with various silver halide developing agents. Suitable silver halide developing agents include any which do not adversely affect the fixing and/or silver halide solvent activity of the β -disulfones. Suitable silver halide developing agents which can be employed include, for example, gallic acid, p-phenylene diamines, polyhydroxybenzenes, such as hydroquinone developing agents, e.g. hydroquinone, alkyl-substituted hydroquinones, as exemplified by tertiary butyl hydroquinone and methyl

hydroquinone; catechol and pyrogallol; chloro-substituted hydroquinone such as chlorohydroquinone or dichlorohydroquinone; alkoxy-substituted hydroquinone such as methoxy hydroquinone or ethoxy hydroquinone; aminophenol developing agents such as 2,4-diaminophenols and methyl aminophenols; ascorbic acid developing agents such as ascorbic acid, ascorbic acid ketals, and ascorbic acid developing agents as described, for example, in U.S. Pat. No. 3,337,342 of Green issued Aug. 22, 1967; hydroxyl amine developing agents such as N,N-di(2-ethoxyethyl)hydroxyl amine; 3-pyrazolidone developing agents such as 1-phenyl-3-pyrazolidone and 1-phenyl-4-methyl-4-hydroxymethyl-3-pyrazolidone, including those described in British Patent 930,572 published July 3, 1963; and acyl derivatives of para amino phenol such as described in British Patent 1,045,303 published Oct. 12, 1966. Reductone developing agents can be employed with the described β -disulfones, such as dihydroanhydro reductone developing agents, including dihydroanhydro piperidino hexose reductone, as described in Belgian Patent 749,935 issued July 15, 1970; Belgian Patent 749,330 issued July 15, 1970 and Belgian Patent 746,700 issued Apr. 30, 1970. The described developing agents can be used alone or in various combinations.

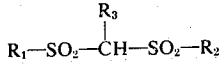
If desired, a wide variety of thickening agents or viscosity-increasing materials can be added to monobath compositions comprising the silver halide solubilizing agents of the present invention in order to impart the high viscosity thereto. High viscosity monobath compositions are desirable as they can be readily spread in a uniform layer on the film to be processed. Such viscosity-increasing materials include film-forming materials that retain their viscosity in aqueous alkaline composi-

tions. Suitable viscosity-increasing materials include sodium carboxymethylcellulose, hydroxyethylcellulose, polyvinyl alcohol, sodium salts of polymethacrylic acid and polyacrylic acid, and the like.

The invention has been described in considerable detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications may be effected within the spirit and scope of the invention as described hereinabove and as defined in the appended claims.

We claim:

1. A process for solubilizing unexposed and undeveloped silver halide on a photographic element which comprises treating said silver halide with an aqueous alkaline composition comprising a 1,1 bis-sulfonyl alkane silver halide solubilizing agent represented by the formula



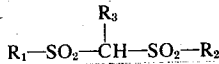
wherein R₁ and R₂ each represent an alkyl group containing one to four carbon atoms and R₃ represents H or an alkyl group containing one to four carbon atoms.

2. The process of claim 1 wherein R₁ and R₂ each represent methyl, ethyl, hydroxyethyl, carboxymethyl, or carboxyethyl groups and R₃ represents H.

3. The process of claim 1 wherein R₁ and R₂ each represent a methyl group.

4. The process of claim 1 wherein said solubilizing agent is present in an amount of from about 3 to about 200 grams per liter of composition.

5. An aqueous alkaline photographic composition comprising a silver halide solubilizing agent having the formula



wherein R₁ and R₂ each represent an alkyl group containing one to four carbon atoms and R₃ represents H or an alkyl group containing one to four carbon atoms.

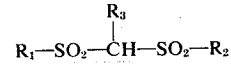
6. A photographic composition as defined in claim 5 wherein R₁ and R₂ each represent methyl, ethyl,

hydroxyethyl, carboxymethyl, or carboxyethyl groups and R₃ represents H.

7. A photographic composition as defined in claim 6 wherein R₁ and R₂ each represent a methyl group.

8. A photographic composition as defined in claim 5 wherein said solubilizing agent is present in an amount of from about 3 to about 200 grams per liter of composition.

9. An aqueous alkaline composition comprising a silver halide developing agent and a silver halide solubilizing agent having the formula



wherein R₁ and R₂ each represent an alkyl group containing one to four carbon atoms and R₃ represents H or an alkyl group containing one to four carbon atoms.

10. The aqueous alkaline composition as defined in claim 9 wherein R₁ and R₂ each represent methyl, ethyl, hydroxyethyl, carboxymethyl, or carboxyethyl groups and R₃ represents H.

11. The aqueous alkaline composition as defined in claim 10 wherein R₁ and R₂ each represent methyl.

12. The aqueous alkaline composition as defined in claim 11 wherein the silver halide developing agent is hydroquinone.

13. The aqueous alkaline composition as defined in claim 9 wherein said solubilizing agent is present in an amount of from about 3 to about 200 grams per liter of composition.

14. The aqueous alkaline composition as defined in claim 9 which also includes a viscosity-increasing material.

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