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(54) Color imaging material and color imaging process using the same.

(57) Construction:

A color imaging material having a substrate and at least one photosensitive layer provided on the substrate, said photosensitive layer comprising, in 100 parts by weight of a binder, 1 to 100 parts by weight of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 430 to 480 nm in color developed condition, 1 to 100 parts by weight of a fulgide compound having photochromic characteristics such that it has an-absorption maximum in the region of 500 to 550 nm in color developed condition, and 1 to 100 parts by weight of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 600 to 650 rim in color developed condition.

Technical Effect:

A full color image can be obtained by a single time exposure. No other processing, such as development or fixation, is required. Multicolor images obtainable according to the invention have good dark-place storage stability and high image quality. The invention permits a wide range of binder selection. According to the invention, an image material capable of repetitive use is provided.

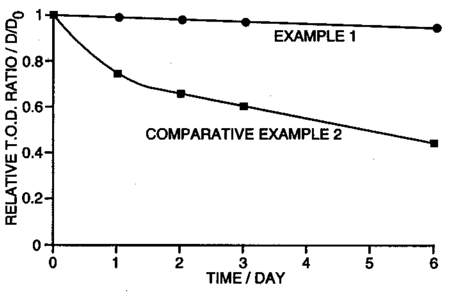


Fig. 5

EP 0 657 773 A1

Rank Xerox (UK) Business Services
(3.10/3.09/3.3.4)

DETAILED DESCRIPTION OF THE INVENTION

Art Field Related:

The present invention relates to color image materials and, more particularly to a color imaging material capable of repetitive use.

Prior Art:

Hitherto, techniques for forming a color image on a substrate have been known including electrophotographic process, ink jet process, heat sensitive recording process, and transfer type heat sensitive recording process, which are already in practical application. However, these techniques have problems in operability, that is, complicated apparatus are required; operations for imaging process is conducted to each of colorants used; and operations for development and fixation are required. With these processes, therefore, it is impracticable to obtain images by simple and easy operation. It is also another problem that these processes can only produce permanent image and cannot provide color imaging material capable of repetitive use which is now largely demanded in the art of imaging.

Attempts at providing a color imaging material capable of repetitive use which permits a color image to be formed in a simple and easy manner have been made by using photochromic compounds which will reversibly be developed and bleached through thermal or luminous energy.

Japanese Patent Kokai Publication No. S59-215382, for example, there is proposed a photochromic photosensitive material which contains at least two kinds of spiropyran having different absorption wavelengths in color developed condition.

In this technique, however, since spiropyran is used as a photochromic compound, any resulting image has poor shelf stability, is of relatively lower resolution, and lacks color clearness. It is another problem that a desired color cannot be obtained unless the spiropyran is dispersed in a particular binder. This does not permit the spiropyran and a freely selected binder to be mixed, and makes it difficult to effect color adjustment. As a result, in order to provide a good color image, it is required to formulate each of the spiropyrans to each of different layers. Also, since single layer structure cannot be obtained, the manufacturing process for the image material is made complicated.

Japanese Patent Kokoku Publication No. S53-23699, there is proposed a color image recording material formed in such a way that photochromic compositions having different visible light absorption spectra in colored condition are laid one over another in layer form or uniformly mixed together. However, in this case, spiropyrans are used as photochromic compounds; and a particular combination of the spiropyrans, a matrix in which the spiropyrans are to be dispersed, and additives to be added to the matrix are used to obtain predetermined absorption spectrum distribution characteristics. Therefore, this technique involves the same problems as the above mentioned case.

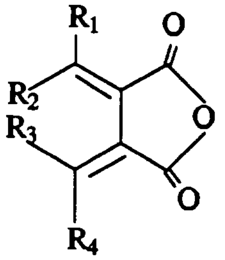
Japanese Patent Application No. H4-227401, there is described a photosensitive material using two kinds of photochromic materials including at least one kind of fulgide. This photosensitive material can be manufactured in a simple process and provides good dark room image storage stability and good image quality. Further, it permits a wider range of binder resin selection. However, the material cannot produce a full color image. As such, an image material capable of repetitive use which can provide a full color image has been eagerly demanded.

Problems to be Solved by the Invention:

The present invention is directed to solving the foregoing problems in the prior art, and accordingly it is an object of the invention to provide a color imaging material which can be manufactured and processed for imaging in a simple and easy manner, and which can develop color images-of high quality and is capable of repetitive use. Means for Solving the Problems:

The present invention provides a color imaging material having a substrate and at least one photosensitive layer provided on the substrate, the photosensitive layer comprising, in 100 parts by weight of a binder, 1 to 100 parts by weight, preferably 10 to 70 parts by weight, of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 430 to 480 nm in color developed condition, 1 to 100 parts by weight, preferably 10 to 70 parts by weight, of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 500 to 550 nm in color developed condition, and I to 100 parts by weight, preferably 10 to 70 parts by weight, of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 600 to 650 nm in color developed condition, whereby the foregoing object can be accomplished.

The photosensitive layer used in the invention comprises a binder having good compatibility with fulgide compounds, the binder containing fulgide compounds having photochromic characteristics in particular wavelength regions. Fulgide compounds useful for the purpose of the invention have a structure expressed by, for example, the following formula:



I

wherein, R_1, R_2, R_3, and R_4 are five-member heterocyclic radicals independently selected from a group consisting of 3-pyrrolyl, 3-indolyl, 3-thienyl, 3-furyl, 4-oxazolyl, 4-thiazolyl, and 4-iso-oxazolyl radicals which can have up to three substituents selected from a group consisting of hydrogen atoms; alkyl radicals having 1 to 6 carbon atoms; aryl radicals having 6 to 7 carbon atoms; alkyl radicals having 1 to 6 carbon atoms, aryl radicals having 6 to 7 carbon- atoms, and alkoxy radicals having 1 to 6 carbon atoms.

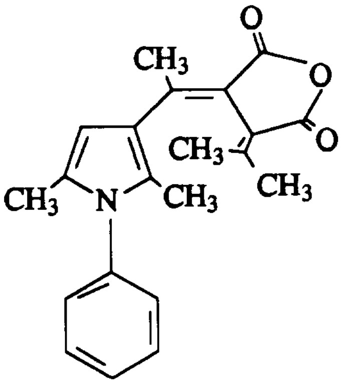
An amount of each fulgide compound added to the binder is 1 to 100 parts by weight, preferably 10 to 70 parts by weight, relative to 100 parts by weight of the binder. If the amount of each fulgide compound added is less than 1 part by weight, the resulting color imaging material will provide insufficient color development. if the amount is greater than 100 parts by weight, there will arise problems, such as decreased mechanical strength of the photosensitive layer, separation of the photosensitive layer from the substrate, and decreased compatibility between the fulgide compound and the binder.

These fulgide compounds differ from each other in absorption spectrum distribution characteristics in their color developed condition. In the present invention, it is preferred that the photosensitive layer contains at least one kind each of fulgide compounds having absorption characteristics corresponding to those of three primary colors (i. e., cyan, magenta, and yellow).

Fulgide compounds having photochromic characteristics in color developed condition are bleached by application of optical energy through exposure to light. From the standpoint of image stability and ease of imaging, fulgide compounds having desirable photochromic characteristics for use in the present invention have a photobleach energy range of 0.1 to 1000 mJ/mm^2, more preferably 1 to 100 mJ/mm^2. For the purpose of image quality improvement, it is preferable to use in combination fulgide compounds having photochromic characteristics which have an even degree of photobleach energy for incorporation of them into the photosensitive layer. Each photochromic compound has different photosensitive wavelengths respectively, and therefore, it can be selectively bleached by the corresponding wavelength of irradiation, it being thus possible to obtain a full color image.

Examples of fulgide compounds which can be advantageously used in the present invention, that is, fulgide compounds having photochromic characteristics which can develop colors corresponding generally to cyan, magenta and yellow, are shown below. Cyan Compounds:

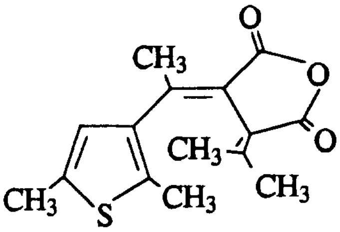
(E)-2-[l-(l-phenyl-2,5-dimethyl-3-pyrrolyl) ethylidene] isopropylidene succinic anhydride; (E)-2-[l(1,2,5-trimethyl-3-pyrrolyl) ethylidene] isopropylidene succinic anhydride; and (E)-2-[l-(1,2-dimethyl-5-methoxy-3-indolyl) ethylidene] isopropylidene succinic anhydride, all of which have a structure expressed by the formula:



II

Magenta Compounds:

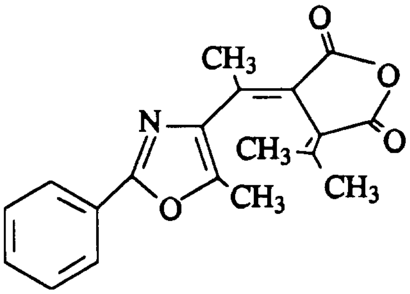
(E)-2-[l-(2,5-dimethyl-3-thienyl) ethylidene] isopropylidene succinic anhydride; (E)-2-[l-(2,5-dimethyl-3-furyl) ethylidene] isopropylidene succinic anhydride; and (E)-2-[l-(2.5-dimethyl-3-furyl) ethylidene]-2-adamanthilidene succinic anhydride, all of which have a structure expressed by the formula:



III

Yellow Compounds:

(E)-2-[l-(5-methyl-2-phenyl-4-oxazolyl) ethylidene] isopropylidene succinic anhydride; (E)-2-[l-(5-methyl-2-phenyl-4-thiazolyl) ethylidene] isopropylidene succinic anhydride; and (E)-2-(l-(3,5-dimethyl-4-isoxazolyl) ethylidene] isopropylidene succinic anhydride, all of which have a structure expressed by the formula:



IV

In the present invention, the photosensitive layer may be comprised of either a single layer or a plurality of layers. Where a photosensitive layer comprised of plural layers is to be formed, it is desirable to provide an isolating layer between the plural layers so that superposed layers will not be mixed together. The isolating layer may be formed by applying a film forming solution using a solvent which will not dissolve the binder resin and photochromic compounds in the photosensitive layer. For example, such a layer can be formed by coating an aqueous solution of polyvinyl alcohol.

For the binder in the photosensitive layer, it is preferred to use a resin of the type which will not adversely affect the photochromism of fulgide compounds having photochromic characteristics, and which has good compatibility with the photochromic compounds, good film-forming capability, and good transparency in its cured state. It is-especially preferable that the binder in its cured state is a transparent material having a Tg of not lower than O°. If the Tg of the cured binder is lower than O°, surface tackiness will occur so that the surface is liable to dust deposition, which is a cause of image quality deterioration. Further, the modulus of elasticity of the photosensitive layer at room temperature will become poor, which results in decreased mechanical strength. Where the binder is not transparent, there will arise problems including decreased color developing capability, decreased sensitivity, and lowered reuse characteristics, of the photosensitive layer.

Examples of useful binder resins include, for example, polystyrene, polyester, methyl polymethacrylate, vinyl chloride, vinylidene chloride copolymers, polyvinyl chloride, polyvinylidene chloride, polyvinyl acetate, and cellulose acetate.

For the substrate on which the photosensitive layer is formed may be used all kinds of materials known to those skilled in the art. For example, transparent polymeric substrate materials, such as polyethylene terephthalate, and non-transparent substrate materials, such as paper, are mentioned. Substrates which are treated and/or primer coated so as to increase adhesion between the photosensitive layer and the substrate can also be advantageously used.

The color imaging material of the present invention can be produced in such a simple and easy way that fulgide compounds having photochromic characteristics and a binder which have been suitably selected are dissolved in a suitable solvent and the resulting solution is coated on the surface of the substrate according to the conventional procedure, followed by the step of drying and film forming. The solvent for use in this connection is not particularly limited insofar as it is able to solve both the fulgide compounds having photochromic characteristics and the binder resin. For example, toluene, methyl ethyl ketone, chloroform, tetrahydrofuran, and ethyl acetate may be mentioned as such.

In order to provide a full color image onto the color imaging material obtained in the above described way, the color imaging material is first exposed to ultraviolet radiation so that all the fulgide compounds having respective photochromic characteristics contained in the color imaging material are brought into color developed condition. Then, the color imaging material is exposed to light beams having a predetermined wave length distribution range to selectively bleach the fulgide compounds having photochromic characteristics corresponding to their respective absorption spectra. Such selective exposure may be made by, for example, exposing the color imaging material to white light through a color positive film.

A protective layer may be formed on the photosensitive layer. Provision of such a layer results in effective protection of the photosensitive layer against oxidation, chemical changes, and mechanical damage, resulting in considerable improvement in the durability of the color imaging material. For the protective layer, silicone or acrylic resins are preferably used. These resins have good transparency. The protective layer is preferably formed to a thickness of 1 to 100 µm. If the thickness of the protective layer is less than 1 µm, the effect of such layer is substantially lost. If the layer is more than 100 µm thick, the color imaging material is less pervious to light, with the result that the image material is less able to develop color.

EXAMPLES

The following examples are given to further illustrate the present invention. It is understood, however, that the invention is not limited to these examples which are taken to be only exemplary.

Example 1:

Ingredients	Proportion (parts by weight)
(E)-2-[l-(l-phenyl-2,5-dimethyl-3-pyrrolyl) ethylidene]isopropylidene succinic anhydride	2
(E)-2-[l-(2,5-dimethyl-3-thienyl)ethylidene] isopropylidene succinic anhydride	2
(E)-2-[l-(5-methyl-2-phenyl-4-oxazolyl)ethylidene] isopropylidene succinic anhydride	2
polystyrene	10
methyl ethyl ketone	18
toluene	72

A solution was prepared from the above ingredients. The solution was coated on a polyethylene terephthalate film of 1OO µm thick by using a #20 Mayer bar and dried, whereby a color imaging material having a photosensitive layer of about 4, µm thick was obtained. By using a high-pressure mercury lamp (made by Ushio Denki) as a light source, ultraviolet radiation in the vicinity of 366 nm was directed to the image material through an ultraviolet light transmission filter (UV-D 35, UV-D 36C, made by Toshiba Corp.) and a heat-absorbing filter (1 cm, $CuSO_4.5H_2O$ aqueous solution (50 g/1), for 2 minutes. Thus, all the fulgide compounds having photochromic characteristics which were contained in the photosensitive layer were brought into color developed condition. A visible absorption spectrum of the color imaging material in such a condition are shown in FIG 1.

The fulgide compounds having three kinds of photochromic characteristics which are contained in the color imaging material were then independently dispersed in polystyrene for being formed into separate single color image materials. These materials were exposed to ultraviolet radiation under the same conditions as above noted. Ultraviolet light and visible light absorption spectra of these materials are shown in FIGS. 2, 3 and 4.

Next, an original color negative slide was placed on the color developed color imaging material, and the composite was exposed to visible light using a 500 W xenon lamp (made by Ushio Denki) and through a UV light cut filter (L-39 S, made by Toshiba Corp.), a sharp cut filter (Y-43, made by Toshiba) and a heat absorption filter (5 cm, water). As a result, photobleach occurred corresponding to the colors of the original color positive slide, and thus a clear full color image was obtained. The color imaging material on which an image was formed was allowed to stand at a dark location under room temperature conditions. Transmission optical density (T.O.D.) changes with time were measured, on the basis of which measurement was evaluated the storage stability of the color imaging material. The results are graphically shown in FIG. 5.

The color imaging material formed with the image was fully exposed to while light for 3 minutes by using a xenon lamp as a light source, so that it was completely bleached and rendered colorless. The color imaging material thus regenerated was used in same way as above described, it being thus possible to form a full color image on it. Repetitive imaging was possible up to 30 times maximum until a formed image was judged unacceptable.

Example 2:

Ingredients	Proportion (parts by weight)
Composition A	
(E)-2-[l-(2,5-dimethyl-3-thienyl)ethylidene] isopropylidene succinic anhydride	1
polystyrene	10
methyl ethyl ketone	18
toluene	72
Composition B	
polyvinyl alcohol	1
water	10
Composition C	
(E)-2-[l-(l-phenyl-2,5-dimethyl-3-pyrrolyl) ethylidene] isopropylidene succinic anhydride	1
polystyrene	10
methyl ethyl ketone	18
toluene	72
Composition D	
(E)-2-[l-(5-methyl-2-phenyl-4-oxazolyl) ethylidene] isopropylidene succinic anhydride	1
polystyrene	10
methyl ethyl ketone	18
toluene	72

A solution of the above composition A was prepared. The solution was coated on a polyethylene terephthalate film of 100 µm thick using a #20 Mayer bar of and was dried. Subsequently, composition B, composition C, composition B, and composition D were similarly coated on the dried layer in that order (except that composition B was coated using #10 Mayer bar), and dried, with the result that a multistructure image material was obtained.

In the same manner as Example 1, the image material was color developed, and then was exposed to visible light using an original color negative slide. As a result, photobleach was occurred corresponding to the colors of the original slide. Thus, a clear full color image was obtained. As was the case with the image material in Example 1, the process of color imaging could be repetitively carried out.

Example 3:

Ingredients	Proportion (parts by weight)
(E)-2-[l-(l-phenyl-2,5-dimethyl-3-pyrrolyl) ethylidene] isopropylidene succinic anhydride	2
E)-2-[l-(2,5-dimethyl-3-furyl)ethylidene] isopropylidene succinic anhydride	2
(E)-2-[l-(5-methyl-2-phenyl-4-oxazolyl) ethylidene] isopropylidene succinic anhydride	2
polystyrene	10
methyl ethyl ketone	18
toluene	72

A solution was prepared from the above ingredients, and an image material was prepared in same way as in Example 1. The obtained image material was color developed in the same manner as Example 1. Then, the image material was subjected to visible light exposure using an original color negative slide. As a result, photobleach occurred corresponding to the colors of the original slide, and thus a clear full color image was obtained. The process of imaging was repetitively carried out as is the case with the image material in Example 1.

Comparative Example 1:

Ingredients	Proportion (parts by weight)
E)-2-[l-(2,5-dimethyl-3-thienyl) ethylidene]isopropylidene succinic anhydride	2
(E)-2-[l-(5-methyl-2-phenyl-4-oxazolyl) ethylidene] isopropylidene succinic anhydride	2
polystyrene	10
methyl ethyl ketone	18
toluene	72

A solution was prepared from the foregoing ingredients, and an image material was prepared according to the same procedure as was used in Example 1. The image material obtained was color developed in same manner as Example 1. Then, visible light exposure was carried out using an original color positive slide. As a result, a multicolor image was formed but no full color image could be obtained.

Comparative Example 2:

Ingredients	Proportion (parts by weight)
Composition A	
6'-nitro-1,3,3-trimethyl spiro[2H-l-benzopyran-2.2'-indoline]	1
cellulose acetate	5
methyl ethyl ketone	50
cyclohexanone	50
Composition B	
polyvinyl alcohol	1
water	10
Composition C	
6-nitro-l'-methyl-3,8-dimethoxy spiro[2H-1-benzopyran-2,2'-benzothiazoline]	3
vinyl chloride-vinylidene chloride copolymer	10
tetrahydrofuran	50
cyclohexanone	50

A solution of the above composition A was prepared. The solution was coated on a polyethylene terephthalate film of 100, µm thick by using a #20 Mayer bar, and was dried.

Subsequently, in the same manner, solutions of composition B and composition C were sequentially coated on the previous coating in that order and was dried. Thus, a multilayer color material was obtained.

The obtained photosensitive material was subjected to exposure to ultraviolet radiation from the composition C layer side, for 4 minutes. Then, in same way as in Example 1, visible light exposure was effected using an original color positive slide, thereby to form an image. The color imaging material on which the image was formed was allowed to stand in a dark room under ordinary temperature conditions. Changes with time in transmission optical density (T.O.D.) were measured. On the basis of these measurements, the image material was evaluated with respect to its storage stability. The results are graphically shown in Fig. 5.

FIG. 5 shows that the color image obtained in Comparative example 2 was extremely poor in dark room storage stability such that in only one day period there was a noticeable decrease in its T.O.D., say, as high as 25 % or more. In contrast to this, the color imaging material of the invention obtained in Example I exhibited good storage stability, or in terms of decrease in T.O.D. in full six days, there was only 5% decrease.

Technical Effect of the Invention:

A full color image can be obtained by a single exposure. No other processing, such as development or fixation, is required. Multicolor images obtainable according to the invention have good dark-place storage stability and high image quality. The invention permits a wide range of binder selection. According to the invention, an image material capable of repetitive use is provided. Brief Description of the Drawings:

FIG. 1 shows a visible light absorption spectrum of a color imaging material according to the invention as seen in color developed condition;

FIG. 2 shows a visible light absorption spectrum with respect to one kind of fulgide compound having photochromic characteristics contained in the color imaging material as seen in color developed condition;

FIG. 3 shows a visible light absorption spectrum with respect to one kind of fulgide compound having photochromic characteristics contained in the color imaging material as seen in color developed condition;

FIG. 4 shows a visible light absorption spectrum with respect to one kind of fulgide compound having photochromic characteristics contained in the color imaging material as seen in color developed condition; and

FIG. 5 shows a graph showing the storage stability of an image formed on a color imaging material of the present invention.

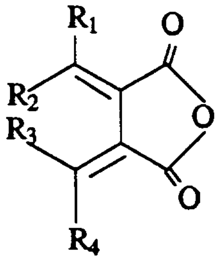
Claims

1. A color imaging material having a substrate and at least one photosensitive layer provided on the substrate, said photosensitive layer comprising, in 100 parts by weight of a binder, 1 to 100 parts by weight of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 430 to 480 nm in color developed condition, 1 to 100 parts by weight of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 500 to 550 nm in color developed condition, and 1 to 100 parts by weight of a fulgide compound having photochromic characteristics such that it has an absorption maximum in the region of 600 to 650 nm in color developed condition.

2. A color imaging material as set forth in claim 1, wherein the photosensitive layer contains, in 100 parts by weight of the binder, 10 to 70 parts by weight of the fulgide compound having photochromic characteristics which has an absorption maximum in the region of 430 to 480 nm in color developed condition, 10 to 70 parts by weight of the fulgide compound having photochromic characteristics which has an absorption maximum in the region of 500 to 550 nm in color developed condition, and 10 to 70 parts by weight of the fulgide compound having photochromic characteristics which has an absorption maximum in the region of 600 to 650 nm in color developed condition.

3. A color imaging material as set forth in claim 1, wherein the fulgide compounds having photochromic characteristics have a structure expressed by the formula:

I



wherein, R,, R,, Ra, and R. are independently selected from a group consisting of 3-pyrrolyl, 3-indolyl, 3-thienyl, 3-furyl, 4-oxazolyl, 4-thiazolyl, and 4-isooxazolyl radicals which can have up to three substituents selected from a group consisting of hydrogen atoms; alkyl radicals having 1 to 6 carbon atoms; aryl radicals having 6 to 7 carbon atoms; alkyl radicals having 1 to 6 carbon atoms, aryl radicals having 6 to 7 carbon atoms, and alkoxy radicals having I to 6 carbon atoms.

4. A color imaging material as set forth in any one of claims 1 to 3, wherein the binder, in its cured state, is a transparent material having Tg of not less than 0 ° C.

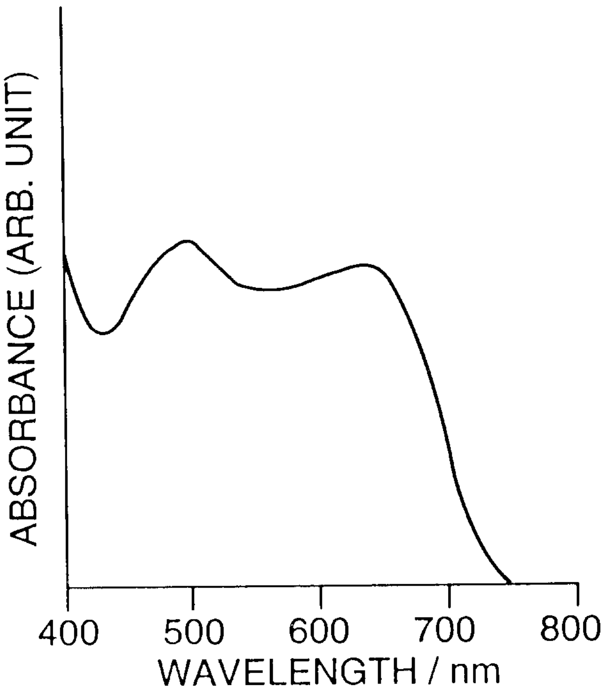
5. A color imaging material as set forth in any one of claims I to 4, wherein the photosensitive layer is 1 to 100,u m thick.

6. A color imaging material as set forth in any one of claims 1 to 5, wherein on the surface of the photosensitive layer there is provided a protective layer formed of a silicone resin or acrylic resin.

7. A color imaging material as set forth in claim 6, wherein the protective layer is 1 to 100 μm thick.

8. A color imaging process comprising the steps of:
  a) exposing the color imaging material defined in claim 1 to ultraviolet radiation thereby to develop all the fulgide compounds having photochromic characteristics which are contained in the photosensitive layer; and
  b) selectively bleaching the developed fulgide compounds having photochromic characteristics in accordance with a desired pattern and in a desired wavelength region.

9. A method as set forth in claim 8, wherein the bleaching step is carried out by projecting white light through a color positive film.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WAVELENGTH / nm

Fig. 1

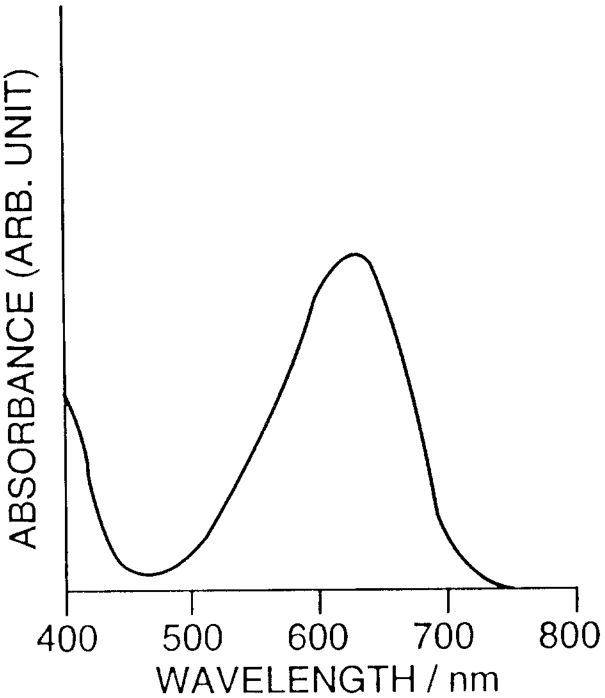
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Fig. 2

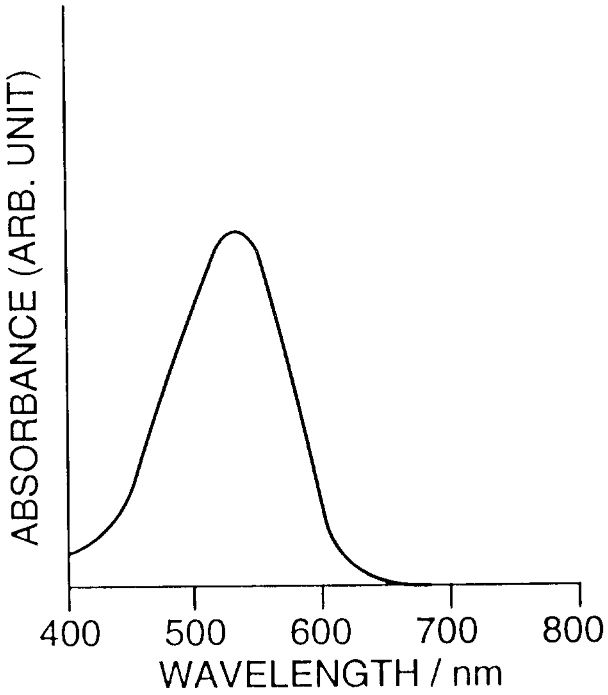
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Fig. 3

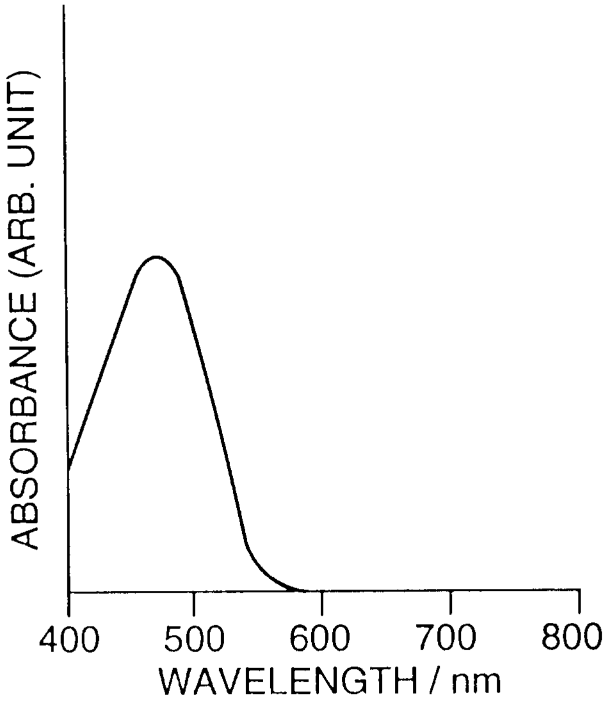
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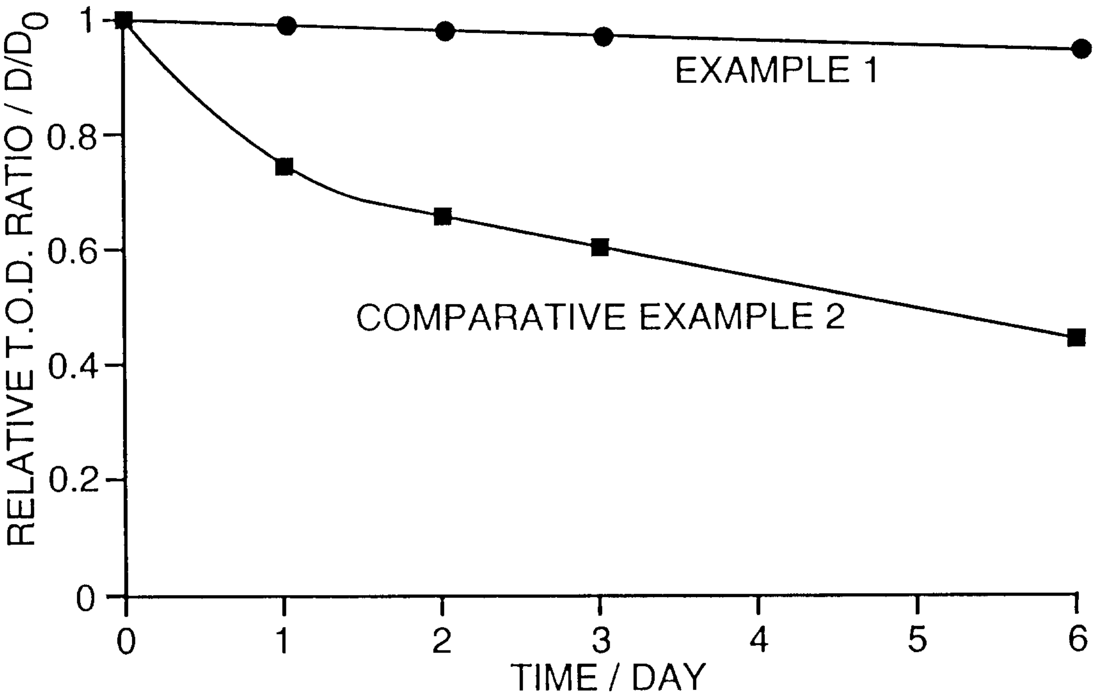
Fig. 4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TIME / DAY
EXAMPLE 1
COMPARATIVE EXAMPLE 2

Fig. 5

European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 94 11 9246

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	US-A-2 186 942 (O VIERLING) * page 2, column 2, line 3 - line 21 * ---	1-4,8,9	G03C1/73
A	DATABASE WPI Week 8929, Derwent Publications Ltd., London, GB; AN 89-210842 & JP-A-1 148 590 (AGENCY OF IND SCI TECH) * abstract * ---	1,8	
A	US-A-3 909 266 (E INOUE ET AL) * the whole document * ---	1,8	
P,D, A	DATABASE WPI Week 9419, Derwent Publications Ltd., London, GB; AN 94-153936 & JP-A-6 095 291 (MINNESOTA MINING AND MANUFACTURING) * abstract * -----	1,8	

TECHNICAL FIELDS SEARCHED (Int.Cl.6)

G03C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	14 February 1995	Heywood, C

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