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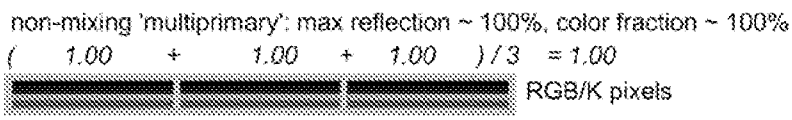
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(54) Title: SINGLE FORMULATION, 4-COLOR RGBK, QUASI-PHOTONIC CRYSTAL BASED REFLECTIVE DISPLAYS

Figure 3



(57) Abstract: Disclosed are formulations for and processes for making reflective displays. The formulations and processes include the addition of an absorptive filler, such as carbon black, to produce all the colors necessary to produce a full-color reflective display.

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SINGLE FORMULATION, 4-COLOR RGBK, QUASI-PHOTONIC CRYSTAL BASED REFLECTIVE DISPLAYS

Cross-Reference to Related Applications

This application claims benefit of U.S. Provisional Application Serial No. 61/740,167, filed December 20, 2012, the disclosure of which is incorporated herein by reference in its entirety.

Field of the Invention

This invention relates to quasi-photonic crystal based color-capable reflective displays using a single formulation. More specifically, this invention relates to reflective displays using both reflective and absorptive particles, which are capable of producing all of the colors necessary to produce a full-color reflective display.

Background

Photonic crystals can be used for display devices. An electric field is applied to particles having electric charges or electrical polarization characteristics and/or a solvent with electrical polarization characteristics to control inter-particle distances of the particles and thereby reflect light of a certain wavelength range from the particles.

Color models require that a reflective display not only be able to produce a colored state, i.e., red (R), green (G), or blue (B), but also produce black (K) and white (W) in order to display the full range of colors perceived by the human eye. The existing display technologies are capable of producing colored states, i.e., R, G, and B, or black and white colored states, i.e., K or W, but not both. Full color is then achieved by creating a hybrid device by stacking multiple two state components in the normal to the viewing plane and assembling multiple states side by side within the plane (*See* Figure 1). The term hybrid is used loosely as a RGB/transparent

material with a black background that would satisfy the RGBK color architecture requirements.

The problem with these stacked reflective displays is that the layers above reduce the reflectivity from each layer, and thus achieving proper color balance is difficult. Horizontal hybrids use two or more technologies to achieve the necessary RGBK color architecture, where one technology achieves RGB and another in parallel achieves BW. If this is done at sufficiently small size, the human eye perceives this as a single color. This mixing of technologies horizontally in a display adds considerable complexity, increasing the rate of defects and manufacturing costs.

The need to mix these colors in a hybrid device is driven by color theory, which describes the maximum color gamut available due to the mixing of multiple colors. A summary of how R, G, and B colors can be used to make multiple colors for different display technologies is shown in Figure 2. The different triangles within the plot indicate the maximum number of colors available to the different display technologies. For example, the NTSC standard color gamut is capable of producing a red, green, and blue, or by appropriate mixing of the intensities of each of those colors, produces all colors within the triangle. The colors available from mixing of R, G, or B include W, as shown near the center of the color gamut triangle. Noticeably absent is K. In the emissive technologies shown in Figure 1, K is produced by turning off all the colored states to reveal a K colored background. Reflective displays must also have the capability of producing a K color state, but do not have the option of producing such a state by "turning-off." Instead, reflective displays must enable a K state in addition to the R, G, and B. Alternatively, the complementary CMY color set may be used in place of the RGB color set if the reflective display uses

absorption rather than scattering as the primary mechanism for reflecting different colors to the observer.

The addition of absorptive fillers into photonic quasi-crystal reflective displays enables the production of the necessary RGBK color set required to produce a full-colored display. Photonic quasi-crystal colloids rely upon short-range structural color phenomena. In this approach, color is observed by creating a formulation of monodisperse nanoparticles (dia. 100 – 200 nm) and applying an electric field. This electric field drives the particles together using electromigration until electrostatic repulsion counteracts the movement, reaching an equilibrium. The short-range order that develops within the monodisperse nanoparticles selectively allows only one wavelength to reflect back towards the observer. This assumes a diffuse lighting source, as a spatially coherent light source, which strikes the surface at a single angle, will result in angle dependent reflection. A startup in Korea, Nanobrick, demonstrated this concept in a recent publication using silica over iron oxide nanoparticles in propylene carbonate between two indium tin oxide slides (*See* United States Patent Application Nos. 2011023516 and 20120044128; Lee et al., *Quasi-Amorphous Colloidal Structures for Electrically Tunable Full-Color Photonic Pixels with Angle-Interdependency*, Adv. Mater. 2010, 22, 4973-4977). However, Nanobrick demonstrated only three colors, i.e., R, G and B. Thus, they fall short of fulfilling the necessary requirements to produce a full-colored display. A need exists, therefore, for a single formulation reflective display that is capable of producing both color states and black and white states.

Another problem with these displays is the ability to control diffuse white scattering, which is caused by reflection of non-desired wavelengths of light reflecting back out from the surface of the display. Previous attempts to control diffuse white

scattering in photonic crystals rely on absorptive cores within the particles. Such particles allow for the dominant wavelength to scatter back (based on the interparticle distance), while absorbing diffuse scattering within the absorptive cores. However, this approach limits the lightness of the display to a single value, where lightness is defined in accordance with the CIE Lab color model definition. A need exists, therefore, for control of the lightness value without sacrificing the ability to control the dominant reflected wavelength.

Summary

In one aspect, an illustrative embodiment provides a reflective display formulation, comprising a solvent, an absorptive material, and a reflective material.

In another aspect, an illustrative embodiment provides a reflective display comprising a plurality of pixels, wherein each of the plurality of pixels comprise a solvent, an absorptive material, and a reflective material.

In yet another aspect, an illustrative embodiment provides a method of making a reflective display, comprising providing a dispersion comprising a solvent and a plurality of particles dispersed in the solvent, wherein the plurality of particles comprise a reflective material. The method further comprises adding an absorptive material to form a reflective display formulation and filling a plurality of pixels with the reflective display formulation.

The foregoing summary is illustrative only and is not intended to be in any way limiting. In addition to the illustrative aspects, embodiments, and features described above, further aspects, embodiments, and features will become apparent by reference to the drawings and the following detailed description.

Brief Description Of The Drawings

The patent or application file contains at least one drawing executed in color. Copies of this patent or patent application publication with color drawing(s) will be provided by the Office upon request and payment of the necessary fee.

Figure 1 is a drawing of theoretical color architecture options for full color reflective displays from Heikenfeld et al., *Review Paper: A critical review of the present and future prospects for electronic paper*, Journal of the SID, 19/2, 129-156 (2011).

Figure 2 is a CIE Lab color mixing chart described by Tomi Koskela, *LED Backlighting for LCs Requires Unique Drivers*, Power Electronics Technology, May 12, 2008

Figure 3 is a schematic diagram of a proposed color architecture for a system with RGBK capabilities within a single formulation. :

Figure 4 is a photograph showing colors produced from quasi-photonic crystals in acetonitrile (a) with and (b) without absorptive fillers.

Figure 5 is a photograph showing colors produced from quasi-photonic crystals in propylene carbonate (a) with 0.002 weight percent carbon black, and (b) with 0.02 weight percent carbon black.

Figure 6 is a photograph showing colors produced from 110 nm commercial silica in water (a) with 0.01 weight percent carbon black, and (b) without carbon black.

Figure 7 is a photograph showing colors produced from quasi-photonic crystals in propylene carbonate (a) with 0.002 weight percent carbon black, (b) 0.01 weight percent, and (c) with 0.02 weight percent carbon black.

Figure 8 is a photograph showing colors produced from 110 nm commercial silica particles, carbon black, and propylene carbonate at various concentrations of silica, and two different carbon black particle sizes.

Figure 9 is a photograph showing colors produced from 134 nm silica particles synthesized by the inventors, with carbon black at 0.01 weight percent and propylene carbonate as a function of silica concentration.

Detailed Description

A single reflective display formulation is provided, which may produce a colored state, i.e., red (R), green (G), or blue (B) and produce black (K) and white (W) in order to, in certain embodiments, display the full range of colors perceived by the human eye. The reflective display formulation is suitable for use in reflective displays. The reflective display formulation may comprise a solvent, an absorptive material, and a reflective material. A particularly suitable reflective material is a material that is capable of reflecting light when used in a reflective display. In a particular embodiment, the reflective display formulation may comprise first particles and second particles, wherein the first particles are reflective material and wherein the second particles are absorptive material. Also in another particular embodiment, the reflective material particles may be present in a concentration and/or size that provides for the perception of color. In an illustrative embodiment, the reflective display may comprise a plurality of pixels. Each of the plurality of pixels may comprise a solvent, an absorptive material, and a reflective material. In another particular embodiment, the reflective display formulation may comprise an absorptive material and a reflective material, wherein the concentrations and sizes of the absorptive material particles and reflective material particles cooperate so that the

formulation, when in a display, is capable of reflecting light to provide for the perception of color.

The solvent may be, for example, acetonitrile, propylene carbonate, dimethyl sulfoxide, dimethoxyethane, mineral oil, silicon oil, ethanol, water, or blends of solvents, where the solvent blend is in a single phase. The absorptive material may be, for example, carbon black, molybdenum disulfide, black dye, or black pigment. In an illustrative embodiment, the absorptive filler may be Monarch® 1100 carbon black. The absorptive material may also be materials such as SKC Haas' mill base series, such as BM-82, BM-100, BM-102, and BM-8. The reflective material may be, for example, silica, titania, zirconia, ceria, alumina, barium titanate, and blends of metal oxides, such as titanosilicates.

The absorptive material may have a primary particle size between about 1 nm and about 200 nm, preferably between about 5 nm and about 30 nm, and more preferably between about 15 nm and about 20 nm. Primary particle size is the size of an individual particle of the absorptive material. Carbon black particles tend to reversibly agglomerate, thus, aggregate particle size may also be measured. The absorptive material may be present in a concentration of between about 0.001 weight percent and about 0.1 weight percent, preferably between about 0.002 weight percent and about 0.2 weight percent, and more preferably between about 0.005 weight percent and about 0.05 weight percent.

The reflective material may have a particle size of between about 75 nm and about 200 nm, preferably between about 100 nm and about 200 nm, and more preferably between about 100 nm and about 150 nm. The reflective material may be present in a concentration of between about 1 weight percent and about 70 weight percent, preferably between about 2 weight percent and about 50 weight percent,

and more preferably between about 5 weight percent and about 40 weight percent. In another embodiment, the reflective material may be present in a concentration of between about 3 weight percent and about 60 weight percent. The size and/or the concentration of the reflective material may be selected so that the reflective material provides for a perception of color.

The reflective display formulation may also comprise a surface modifier. The surface modifier may be, for example, silica or long-chain alkanes. The surface modifier may be covalently bonded and physically bonded to a surface of the reflective material or a surface of the absorptive material to improve the stability in solution or the mobility under an applied electric field. In addition, the reflective display formulation may also comprise a surfactant. The surfactant may be, for example, Dow Tergitol™ 15-S series surfactants, Tween® surfactants, Shell Neodal surfactants, AkzoNobel Armeen® surfactants, or Rhodia Alkamide® surfactants. The reflective display formulation may also comprise dyes and/or pigments, for example, Fire Red G, Fire Red BL, or Orasol® (all examples of red dyes). The dyes and/or pigments may be of a specific color to provide selective modification of the color intensities across the visible color range by absorbing only specific wavelengths. The dyes and/or pigments may substitute for the absorptive filler or may complement the absorptive filler. The addition of dyes and/or pigments may provide for more subtle modification of the colors, adjusting the balance between reds, greens, and blues.

A method of making a reflective display is also provided. The method may comprise providing a dispersion comprising a solvent and a plurality of particles dispersed in the solvent, wherein the plurality of particles comprise a reflective material. The method may further comprise adding an absorptive material to form a

reflective display formulation and filling a plurality of pixels with the reflective display formulation.

A method for reflecting wavelengths is also provided. The method may comprise dispersing a plurality of reflective particles, wherein the reflective particles are capable of electrophoretic migration. The plurality of reflective particles may be arranged in a short-range order. The capability of the reflective particles to electrophoretically migrate may be caused by an electric field and/or charges present on the reflective particles in system. Both electrophoretic migration and electrical polarization may be important to the tunability of the system, i.e., the ability of the system to change colors and to provide a wide range of colors. The method may further comprise adding an absorptive material to form a reflective display formulation and applying the electric field to the reflective display formulation. By applying the electric field, inter-particle distances between the plurality of particles are controlled and the inter-particle distances between the plurality of particles change, so that the wavelength of light reflected from the particles changes according to the changes of the inter-particle distances. The change in inter-particle distances and thus the wavelength of light reflected impacts the color perceived by the human eye.

EXAMPLES

Formulation

1.5 mL of Nissan Chemical Silica Particles in Isopropanol (IPA-ST-ZL) (colloidal silicon dioxide, 70-100 nm particle size, 30-31% w/w silicon dioxide) is centrifuged at 14,000 g for 30 minutes. The excess isopropanol is removed by pipette, and replaced with solvent, e.g., propylene carbonate. This solution is placed in an ultrasonicator for 10 minutes, after which, the solution is centrifuged at 14,000 g

for 30 minutes. Again, excess propylene carbonate is removed by pipette, and replaced with fresh propylene carbonate. This solution is placed in an ultrasonicator (for example, Fisher Scientific FS30, 100 W output) for 10 minutes. This process is repeated once more for a total of three rinses to remove residual isopropanol and replace it with propylene carbonate. After the final centrifugation, the initial 30 weight percent solution is diluted to various concentrations in propylene carbonate. Then, carbon black, Cabot Monarch® 1100, 15-20 nm average primary particle size, is added such that the carbon black quantity is 0.02 weight percent of the total solution. This solution is placed in an ultrasonicator for 10 minutes to disperse the carbon black. Alternative solvents and carbon black concentrations are also prepared in an analogous manner.

Average Color Evaluation

Photographs of the samples are taken against dark or black backgrounds on a Canon® 1Ti camera under standard laboratory fluorescent lighting. The average color for each sample is measured using a color picker tool within standard image manipulation software. The average color over a circular area is reported. Care is taken not to include edge effects or other image imperfections in the area averaged. The hexadecimal values reported by the software are then converted to LAB using standard mathematical algorithms for color model conversion.

Proposed Device Assembly

A device is assembled in which three primary colors are used in a single subpixel within an electronic display. Further, the addition of carbon black shows the inclusion of the K state is possible within a single pixel of an electronic display. A proposed color architecture for a system with RGBK capabilities within a single formulation is shown in Figure 3.

The addition of absorptive fillers allows for formulation of RGBK capable sub-pixels for quasi-crystalline photonic displays, which in turn enables a new color architecture. This new architecture allows for greatly simplified device fabrication, as only a single active layer is required.

Example 1

Demonstrated in these examples is the addition of absorptive filler, i.e., Cabot Monarch® 1100 carbon black, to colloidal silica, which at sufficiently high concentrations produces scattering from the quasi-crystalline state that forms. The use of high concentration silica simulates the conditions near the electrode under an applied electric field. Shown in Figure 4 is silica in acetonitrile at (a) 0.01 weight percent carbon black and (b) 0 weight percent carbon black at 40, 30, 24, 20, 16, 14, 10, and 6 weight percent silica, left to right. The blue and green state observed in Figure 3(a), with carbon black, is very apparent upon visual inspection. In contrast, the red and black states are muted. This is an inherent challenge for all photonic crystal-based scatters, as the intensity of the scattering is greater at shorter wavelengths, than at longer wavelengths, i.e., more intense in the blues than reds.

Of importance in Figure 4 is the contrast between the (a) with and (b) without carbon black states at the lowest concentration of silica where scattering from the quasi-crystal is negligible. Without carbon black, the diffuse scattering from the silica makes the solution appear white. This works against the required color architecture options described above, as RGBW does not provide the K necessary to produce full colors. With the addition of carbon black, the W state shifts noticeably to a dark grey state.

Example 2

In this example, the acetonitrile is replaced with propylene carbonate. Shown in Figure 5 is silica in propylene carbonate at a) 0.002 weight percent Cabot Monarch® 1100 carbon black and b) 0.02 weight percent carbon black at 40, 30, 24, 20, 16, 14, 10, and 6 weight percent silica, left to right. For the 0.02 weight percent carbon black concentration, the quality of the K state becomes truer. In addition, the R, G, and B state also improve.

Quantifying the color states using the International Commission on Illumination's LAB color model (see Table 1 below) enables a direct comparison between the quality of the K states in each of the photos lowest concentrations. In both the acetonitrile case and the propylene carbonate case, the K state of the sample improved by more than 40% through the addition of a small amount of carbon black.

Table 1: Average LAB Color Values for 6 wt% silica samples in Figures 4 and 5

Sample	Pixels in Sample	HEX	L	A	B
Acetonitrile, 0.00wt% Carbon Black	1963	9d9f99	65	-2	3
Acetonitrile, 0.01wt% Carbon Black	1963	4a505c	34	1	-8
Propylene Carbonate, 0.002wt% Carbon Black	452	697686	49	-1	-10
Propylene Carbonate, 0.02wt% Carbon Black	452	41484e	30	-1	-5

Example 3

In this example, water is used as the solvent instead of acetonitrile or propylene carbonate. Shown in Figure 6 is silica in water at a) 0.01 weight percent Cabot Monarch® 1100 carbon black and b) 0 weight percent carbon black at 40, 30, 24, 20, 16, 14, 10, and 6 weight percent silica, left to right. Figure 6 shows that the

addition of carbon black also improves the color states when water is used as a solvent.

Example 4

This example expands upon Example 2 by including a third carbon black concentration at 0.01 weight percent. Shown in Figure 7 is silica in propylene carbonate at a) 0.002 weight percent carbon Cabot Monarch® 1100 black, b) 0.01 weight percent carbon black, and c) 0.02 weight percent carbon black at 40, 30, 24, 20, 16, 14, 10, and 6 weight percent silica, left to right. Figure 7 shows that increasing the weight percent of carbon black improves the color states.

Example 5

In addition, the type of carbon black has a significant effect on the colors produced from the formulation. Shown in Figure 8 is silica in propylene carbonate at a) 15-20 nm carbon black primary particle size (40-80 nm aggregate particle size (carbon black particles tend to reversibly agglomerate together)) and b) approximately 30-120 nm carbon black primary particle size (approximately 300-700 nm aggregate particle size) at 40, 30, 24, 20, 16, 14, 10, and 6 weight percent silica, left to right. The primary particle size and aggregate particle size are measured by SEM (not shown). As shown in Figure 8, the smaller carbon black particle size has less diffuse scattering.

Example 6

134 nm silica particles are synthesized using the Stöber process, a physical chemistry process for the generation of monodispersed particles of silica. Shown in Figure 9 is 134 nm silica particles, carbon black at 0.01 weight percent, and propylene carbonate as a function of silica concentration, at 5, 8, 10, 15, 20, 30, 40, 50, and 60 weight percent silica, left to right. The colors produced at the same

particle size are significantly different from those of the 110 nm commercial silica despite being of similar morphology.

While the invention has been described above according to its preferred embodiments, it can be modified within the spirit and scope of this disclosure. This application is therefore intended to cover any variations, uses, or adaptations of the invention using the general principles disclosed herein. Further, the application is intended to cover such departures from the present disclosure as come within the known or customary practice in the art to which this invention pertains and which fall within the limits of the following claims.

What is claimed is:

1. A reflective display formulation, comprising a solvent, an absorptive material, and a reflective material.

2. The formulation of claim 1, wherein the solvent is selected from the group consisting of acetonitrile, propylene carbonate, dimethyl sulfoxide, dimethoxyethane, mineral oil, silicon oil, ethanol, water, and blends of solvents in a single phase.

3. The formulation of any one of claims 1-2, wherein the absorptive material is selected from the group consisting of carbon black, molybdenum disulfide, black dyes, and black pigments.

4. The formulation of any one of claims 1-3, wherein the absorptive material is carbon black.

5. The formulation of any one of claims 1-4, wherein the absorptive material has a primary particle size of between 5 nm and 30 nm.

6. The formulation of any one of claims 1-5, wherein the absorptive material is present in a concentration of between 0.002 weight percent and 0.2 weight percent.

7. The formulation of any one of claims 1-6, wherein the reflective material is selected from the group consisting of silica, titania, zirconia, ceria, alumina, barium titanate, and blends of metal oxides.

8. The formulation of any one of claims 1-7, wherein the reflective material has a particle size of between 100 nm and 150 nm.

9. The formulation of any one of claims 1-8, wherein the reflective material is present in a concentration of between 2 weight percent and 50 weight percent.

10. The formulation of any one of claims 1-9, wherein the reflective particles are capable of electrophoretic migration.

11. A reflective display comprising the formulation of any one of claims 1-10.

12. A method of making a reflective display, comprising:

providing a dispersion comprising a solvent and a plurality of particles dispersed in the solvent, wherein the plurality of particles comprise a reflective material;

adding an absorptive material to form the display formulation of any one of claims 1-11; and

filling a plurality of pixels with the reflective display formulation.

13. A reflective display comprising a plurality of pixels, wherein each of the plurality of pixels comprise a solvent, an absorptive material, and a reflective material.

14. A method of making a reflective display, comprising:

providing a dispersion comprising a solvent and a plurality of particles dispersed in the solvent, wherein the plurality of particles comprise a reflective material;

adding an absorptive material to form a reflective display formulation; and

filling a plurality of pixels with the reflective display formulation.

Figure 1

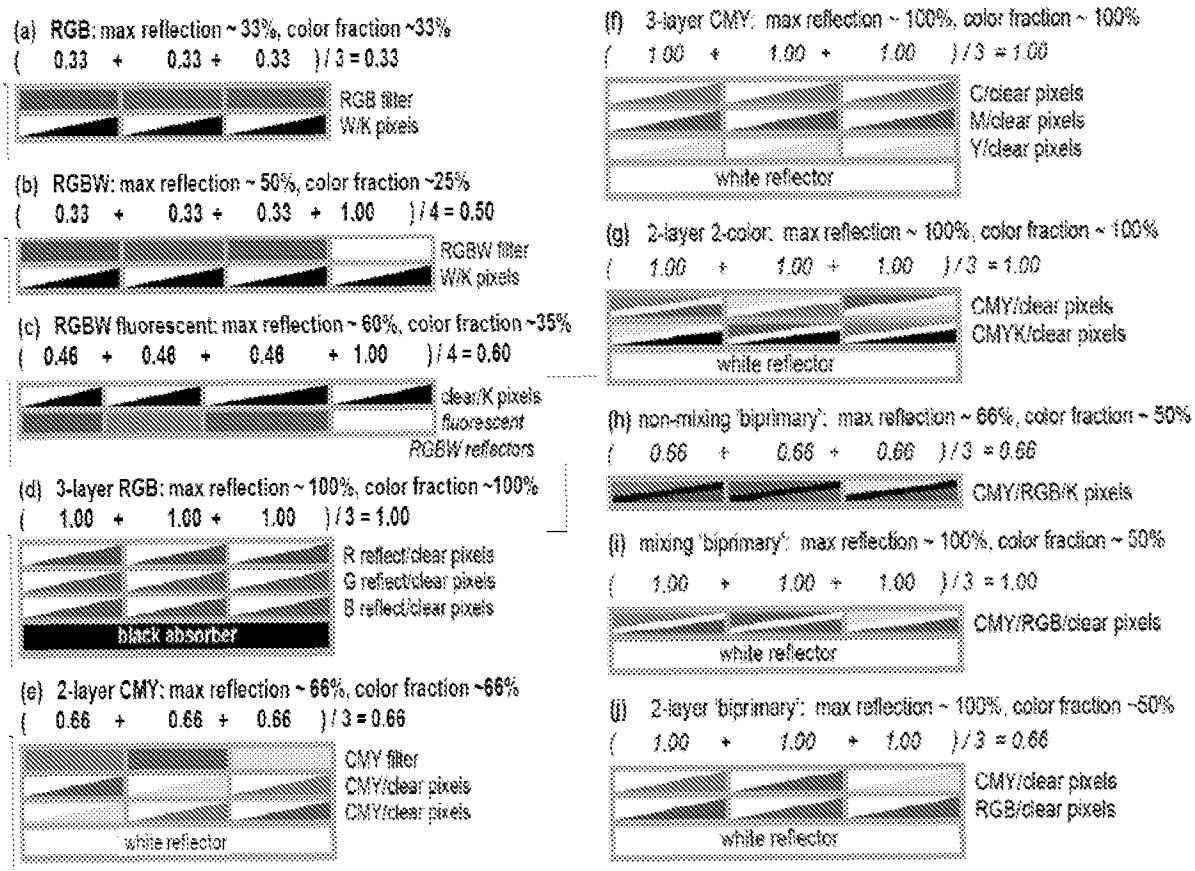


Figure 2

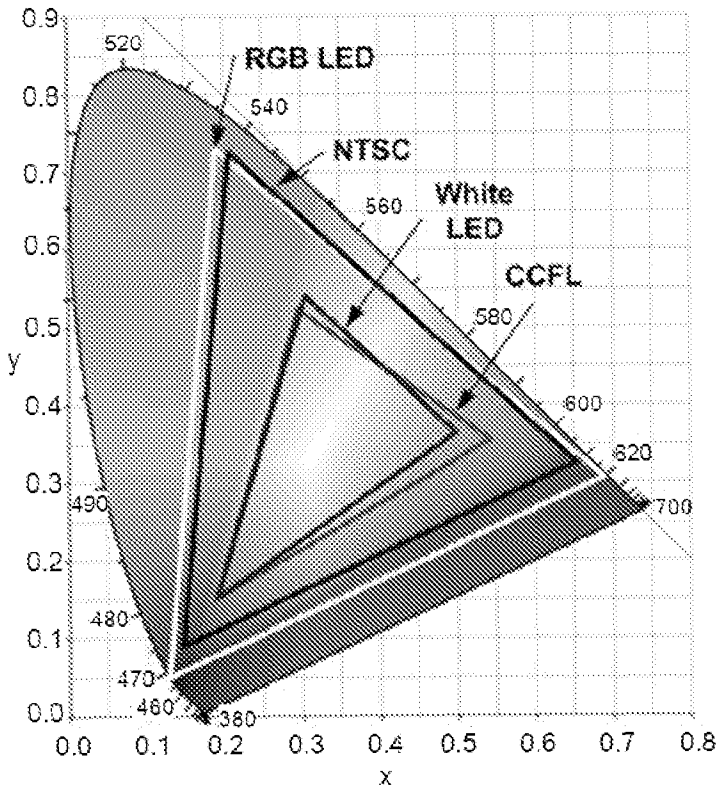


Figure 3

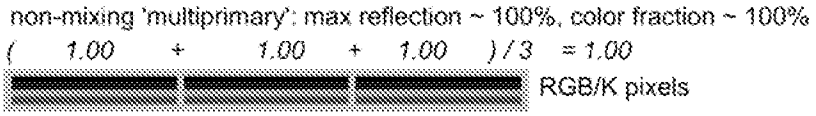


Figure 4

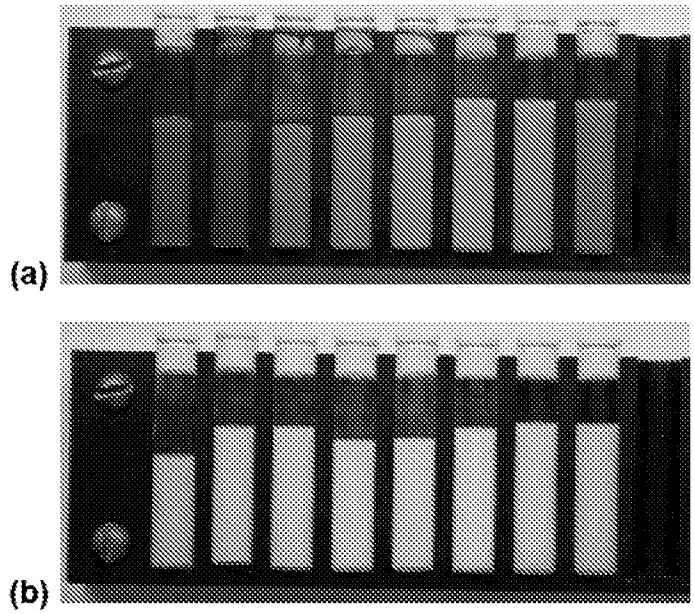


Figure 5:

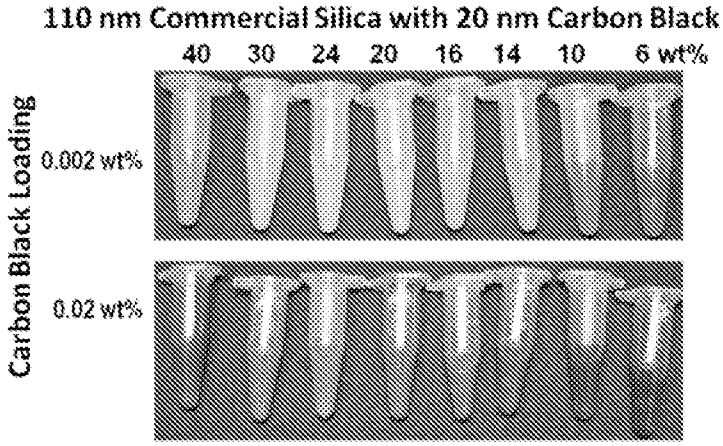


Figure 6

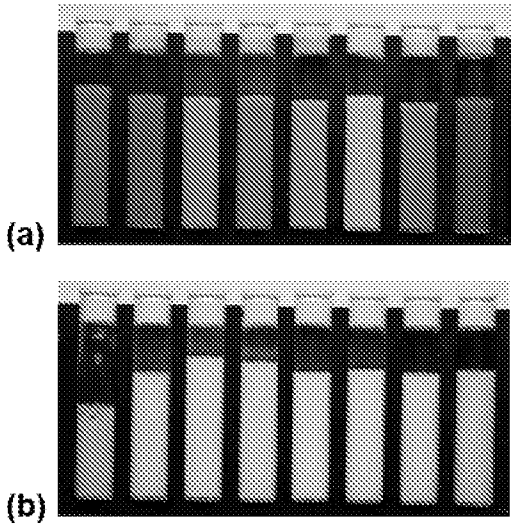


Figure 7

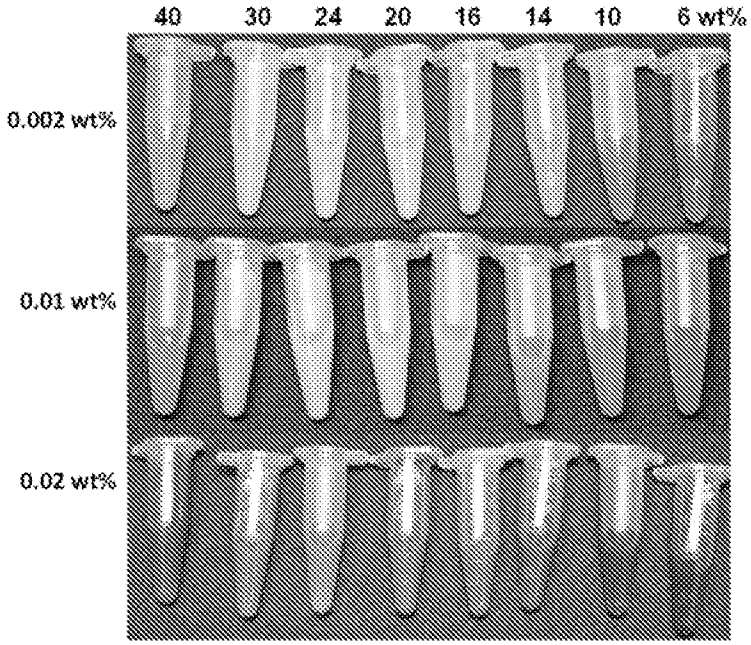


Figure 8

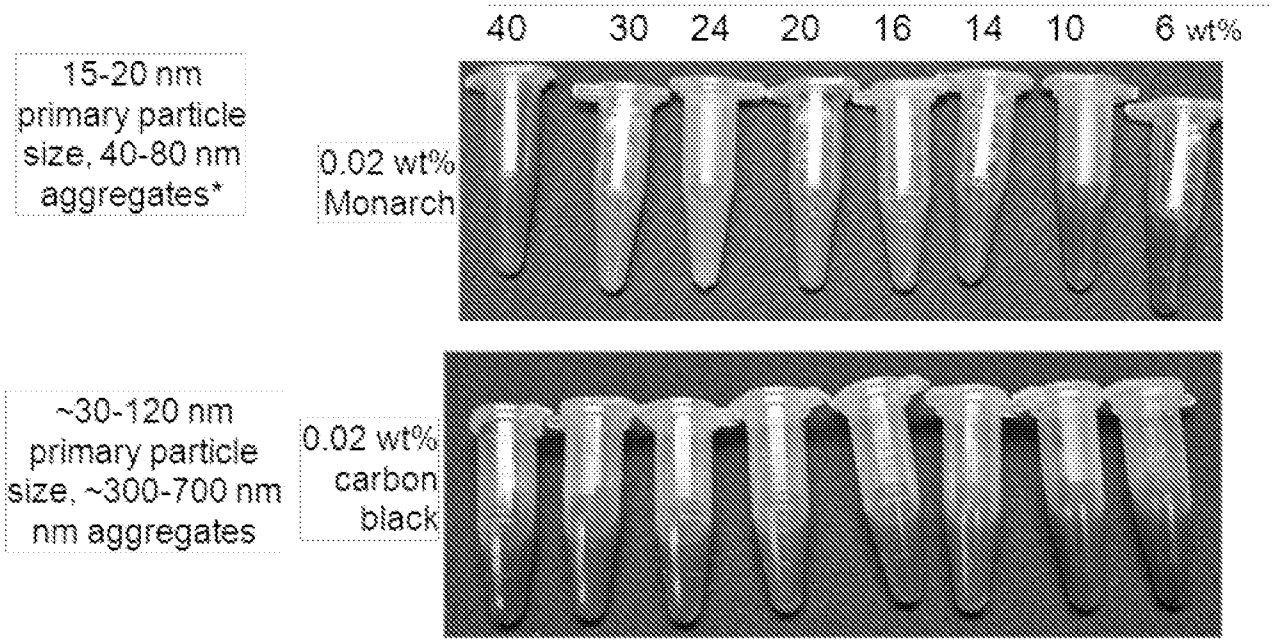
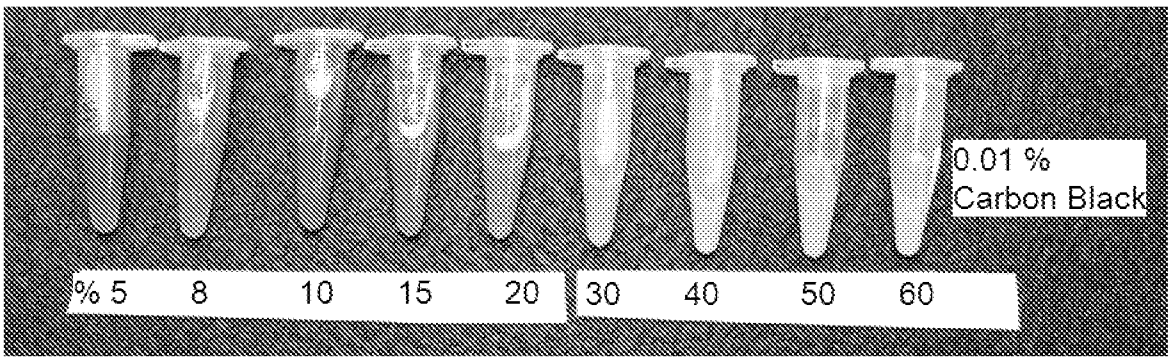


Figure 9



INTERNATIONAL SEARCH REPORT

International application No
PCT/US2013/077022

A. CLASSIFICATION OF SUBJECT MATTER
INV. G02F1/21 G02F1/167 G02F1/19
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
G02F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, COMPENDEX, INSPEC, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 7 259 744 B2 (ARANGO ALEXI C [US] ET AL) 21 August 2007 (2007-08-21) column 4, line 1 - line 50 column 9, line 3 - line 41 column 10, line 53; figures 1,2	1,3,4,7, 10
X	US 2012/293858 A1 (TELFER STEPHEN J [US] ET AL) 22 November 2012 (2012-11-22) paragraphs [0004] - [0007], [0009], [0015], [0021], [0038] - [0040]; figures 2,3	1,11-14
X	KR 2011 0031645 A (NANOBRICK CO LTD [KR]) 29 March 2011 (2011-03-29) abstract paragraphs [0034], [0042] - [0044], [0048] - [0067]; figures 4-9	1,7,8, 10-14

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
15 April 2014	23/04/2014

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Stang, Ingo

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INTERNATIONAL SEARCH REPORT

International application No
PCT/US2013/077022

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	paragraphs [0082], [0084], [0142], [0144]; figure 27	9

Y	INSOOK LEE ET AL: "Quasi-Amorphous Colloidal Structures for Electrically Tunable Full-Color Photonic Pixels with Angle-Independency", ADVANCED MATERIALS, vol. 22, no. 44, 24 November 2010 (2010-11-24), pages 4973-4977, XP055113279, ISSN: 0935-9648, DOI: 10.1002/adma.201001954 page 4974, right-hand column	9

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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