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# Wild Plum: Novel particles of improved optical brightness and fluorescence

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**Synopsis**

A novel compound named *Wild Plum* fluoresces blue, and has been synthesized to camouflage skin imperfections, addressing the market demand for an anti-aging product. Wild Plum imparts optical brightness and fluorescence and can be used as an ingredient in cosmetic formulations. Skin appearance before and after application of Wild Plum compounds demonstrated an improved appearance of skin including a decreased number of wrinkles. When added to makeup, lotions, creams, and powders, Wild Plum conveys the glow of healthy youthful skin, replacing other costly or invasive alternatives such as cosmetic surgery.

## INTRODUCTION

### CURRENT MARKET SITUATION

Cosmetics are personal and highly valued consumable goods. They are critical to a person's identity, helping to create or accentuate beauty and youthfulness. As generations of savvy consumers age, products for concealing age spots, wrinkles, and flaws continue to grow in demand. In response to this need, sales of cosmetics are growing at a 5% yearly pace to $265 billion USD globally and $50 billion in the U.S. (1). Sales of anti-aging skin care products and treatments are growing steadily at approximately 30% a year (2). This demand is driven by aging baby boomers who want to reduce and/or eliminate wrinkles and by younger people hoping to prevent signs of aging.

Our team created a technology that has direct applications to target this market. A report entitled "New Beauty for New Consumers 2009" speaks directly to consumers' desires for

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instant semi-permanent or permanent results, effects that Wild Plum can mimic without the drawbacks of harsh chemicals or procedures (3,4).

### THE PROBLEM

The luminous appearance of skin is associated with health and youth (5). Aging, sun exposure, habitual facial expressions, smoking, and poor hydration lead to skin wrinkles (6). As skin ages, its normal level of fluorescence decreases, coinciding with the decrease in elastin and collagen found in the skin's surface layer. Fluorescence is a key factor directly related to the outward appearance of luster, smoothness, and glow commonly associated with younger skin (5,7). Blue is the dominant autofluorescence color found in the skin, but it begins to diminish as a person ages or as a result of excessive sun damage (5,7,8). This results in an outward appearance of dull, older skin, which lacks luster, smoothness, and glow. Therefore, fluorescence can be used as a marker of photoaged skin.

Current products range from basic foundations and powders to extremely invasive chemical peels and surgeries. Chemical peels and dermabrasion treatments are costly and painful, and can leave skin irritated and problem areas inflamed. Cosmetic surgeries are currently the most costly option, with facelifts and Botox® injections becoming more mainstream (13,14). The cosmetics industry also attempts to combat the appearance of aging by incorporating optically active particles that can interact with light in makeup formulations (9,10). Many are limited to tinted coverups that impart color only. Existing coverup products often cake onto skin, actually accentuating the presence of imperfections in the skin by highlighting blemishes and unevenly collecting in enlarged pores and fine lines. Although they can provide uniform color coverage, they fall short of giving skin a healthy glow. There is a need for a cosmetic adjuvant that can impart luminosity and diffuse reflectance of light and fluorescence, to achieve what makeup alone cannot.

## SCIENTIFIC BACKGROUND

### CURRENT OPTICAL BRIGHTENERS

Optical brighteners have permeated the cosmetics industry. Patent literature shows that coumarins, styryl, microspheres, and microcapsules are incorporated into cosmetic formulations for this purpose (11–13). Most optical brighteners are only UV active, and many fluorescent dyes for cosmetics are water-soluble and will not fluoresce in the solid state. There are only a few substances on the market that can be used in cosmetics that fluoresce in the solid state. For example, LipoLight OAP/PVA (10) fluoresces blue, and other mineral powders fluoresce in a wide range of colors, depending on composition (9).

Wild Plum is a new powder that can be synthesized in a one-pot synthesis with inexpensive starting materials (4). It is a blue fluorescent solid, and the tunability of its organic synthesis allows for the broadening of applications to oil-and-water-based formulas.

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FLUORESCENCE OF SKIN

Fluorescence is observed when light is absorbed at one wavelength and then emitted at a longer wavelength. The glow associated with healthy and young skin results from the fluorescence of skin. Typical wavelengths that are associated with young skin are the blue-light wavelengths of 390 nm for elastin and desmosine and 378 nm for collagen (5,7,8). *In vivo* fluorescence methods (5,14) of normal versus sun-exposed skin demonstrate a dramatic decrease in blue and green fluorescence, believed to be caused by UV light from the sun that inhibits cellular repair (15) and results in injury to skin connective tissue, thus causing a reduction in collagen and elastin (Figure 1) (7,16). Older skin and photo-damaged skin that has been exposed to sunlight over time shows a dramatic decrease in fluorescence emission in the blue and green region of the electromagnetic spectrum. This paper presents the application of a new optical brightening agent named *Wild Plum* that improves the luster and visual appearance of skin.

Wild Plum compensates for the reduced blue fluorescence of aged skin, thereby neutralizing imperfections and leaving a natural, luminous appearance. We formulated Wild Plum with talcum powder in order to take advantage of the "soft focus effect" (17,18) that also improves skin appearance by exploiting the diffuse reflectance of light.

## METHODS AND PROCEDURES

FORMULATION OF FOUNDATION, POWDERS, BLUSHES, CREAMS, ETC.

The formulation was designed to achieve a subtle luminescent glow of the product. Commercial foundation or loose powder was formulated with Wild Plum using a procedure developed at Novel Chemical Solutions (4,19). The formulated products were characterized

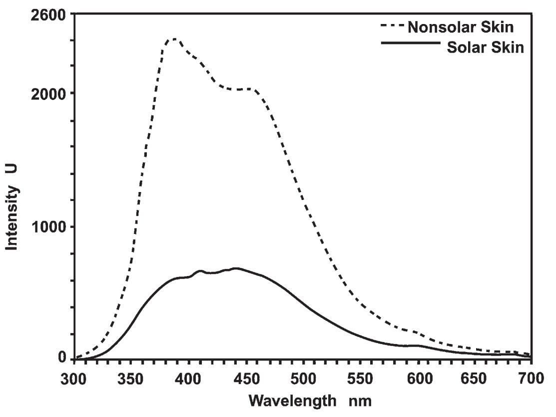


**Figure 1.** Fluorescence emission of sun-exposed skin (forehead) and non-sun-exposed skin (buttocks) demonstrating the dramatic decrease of elastin, desmosine, and collagen fluorescence after sun exposure (at $\lambda_{em}$ = 278 nm, 390 nm, and shoulder at 429 nm). The fluorescence of the skin was induced by a helium-cadmium laser and collected via a fiberoptic probe. (Reprinted with permission from the American Medical Association, *Archives of Dermatology,* 1988, 124, 1514–1518).

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by UV-Vis spectroscopy (Varian Cary 100 UV-visible spectrophotometer), fluorescence spectroscopy (RF-5301PC Shimadzu spectrofluorophotometer), and particle size analysis (Mastersizer X and Hitachi S3000 scanning electron microscope).

### CHARACTERIZATION OF WILD PLUM

*Particle size Analysis.* The particle size was measured with a Hitachi S3000 scanning electron microscope and a Mastersizer X from Malvern Instruments in order to determine if Wild Plum has appropriate granularity for cosmetic applications.

*UV-Vis.* Visible spectroscopy was performed to determine the absorbance profile of Wild Plum. A $4.78 \times 10^{-8}$ M solution of Wild Plum in DMSO was prepared. The sample was placed in a quartz cuvette and measured using a Varian Cary 100 UV-visible spectrophotometer.

*Fluorescence.* A $4.78 \times 10^{-9}$ M solution of Wild Plum in DMSO was prepared for solution fluorescence in order to determine if blue fluorescence emission was achieved in medium-to-high intensity. For solid-state fluorescence, Wild Plum was mixed with talcum powder and placed in a 1-mm quartz cuvette. The cuvette was placed into the sample holder at a 45° angle to the incident beam. The sample was measured using a Shimadzu RF-5301PC spectrofluorophotometer ($\lambda_{ex}$ = 364 nm).

*Quantum yield.* The quantum yield of Wild Plum was measured by the protocol of Jobin Yvon (Horiba), using 9,10-diphenylanthracene and quinine sulfate as quantum yield standards (20).

### *IN VIVO* TESTING OF WILD PLUM

The female model was photographed with a Panasonic DMC-TC1 digital camera under fluorescent office light without makeup, with makeup, and with makeup that was formulated with Wild Plum. L*, a*, and b* values were determined using Adobe Photoshop CS2. This procedure was performed 15 times for each photo, each time selecting color from a different place on the picture and then averaging. Sampling regions were matched between different images.

## EXPERIMENTAL RESULTS

### WILD PLUM FLUORESCENCE

Absorption of light energy excites molecules in these colored materials to their excited states. Fluorescence has occurred when relaxation of these excited states takes place via radiative decay, resulting in emission of light at a longer wavelength than that of the absorbed light. Unlike many other optical brighteners that are active only to ultraviolet light, Wild Plum particles are optically active to visible light and alter the perceived appearance of the skin by emitting favorable light. As seen in Figure 2, this report demonstrates that Wild Plum has a photoluminescence of blue light, with the emission centered between 400 nm and 500 nm ($\lambda_{max}$ = 450 nm). Wild Plum has a fluorescence quantum yield

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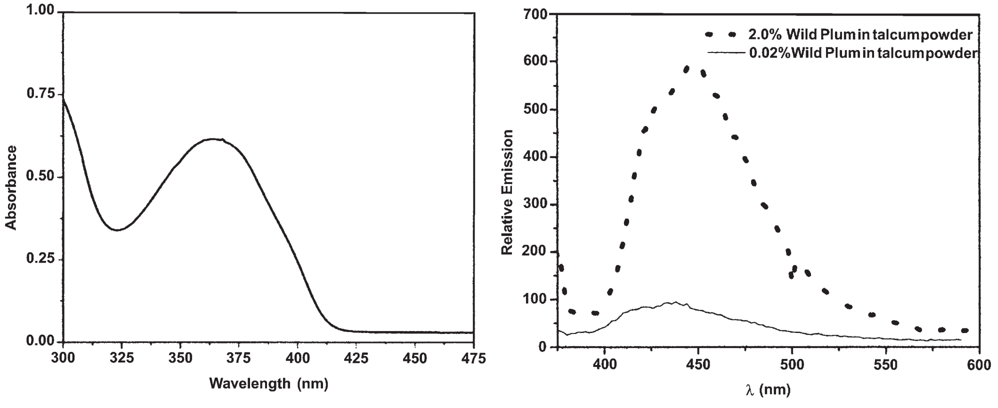


**Figure 2.** (Left) UV-Vis spectrum of Wild Plum at 0.02% in DMSO. (Right) Fluorescence emission spectrum of Wild Plum at 2.0% and 0.02% in talcum powder ($\lambda_{ex}$ = 390 nm; $\lambda_{em}$ = 450 nm).

of 0.29 ± 0.03, allowing for sufficient fluorescence to produce the desired effect without blinding the observer with blue light (data not shown). Wild Plum emits a blue glow similar to the measured blue fluorescence of non-sun-damaged skin (Figure 1), suggesting that Wild Plum has the ability to mimic natural, healthy skin. To the observer, this effect provides an unusual radiance to the skin.

## *IN VIVO* RESULTS

The efficacy of Wild Plum was determined quantitatively by skin color data obtained from the CIELAB color space (21). Color coordinates a*, b*, and L* are considered to correlate with color changes preceived by the human eye. An increase in the L* coordinate indicates lightening and a decrease indicates darkening of the skin surface. Table I shows that L* increases when Wild Plum is added to a commercial foundation. When the a* and b* coordinates increase, reddening and yellowing increase, respectively. Compared to skin without makeup, the addition of Wild Plum to a commercial foundation shows a decrease in red and yellow but an increase in lightness as preceived by the digital camera. Overall, the data demonstrate that luminosity increases while reddening decreases.

*In vivo* data show that Wild Plum quantified the wrinkle condition as we employed a ten-point monadic scale, with one [1] representing the fewest, least prominent fine lines and wrinkles and ten [10] showing the maximum number of deep fine lines and wrinkles.

**Table I**
Skin Color Data Obtained from Adobe Photoshop CS2

| | No makeup | Makeup | Makeup with Wild Plum |
|---|---|---|---|
| L* | 67.4 | 66.5 | 75.8 |
| a* | 16.3 | 19.2 | 15.2 |
| b* | 20.5 | 26.5 | 25.2 |
| | L* Coordinate | a* Coordinate | b* Coordinate |
| Increase | Lightening | Red | Yellow |
| Decrease | Darkening | Green | Blue |

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Application of foundation containing Wild Plum dramatically improved the overall appearance of the skin. Figure 3 shows a 62-year old female without makeup (left), with makeup (center), and with makeup that contains Wild Plum (right).

Figure 4 shows the visual grading wrinkle scores on a ten-point monadic scale (n = 10). The average score for the face image without makeup is 5.6; for the face image with makeup, it is 4.9; and for the face image with makeup containing Wild Plum, it is 2.7. This represents a significant visual appearance of wrinkle decrease. Paired images showing the same part of the face were compared for consistency. In all cases, the difference between no makeup and makeup not containing Wild Plum was not statistically significant

**Figure 3.** A 62-year-old female (left) wearing no makeup; (center) wearing commercial makeup; and (right) wearing commercial makeup formulated with Wild Plum.

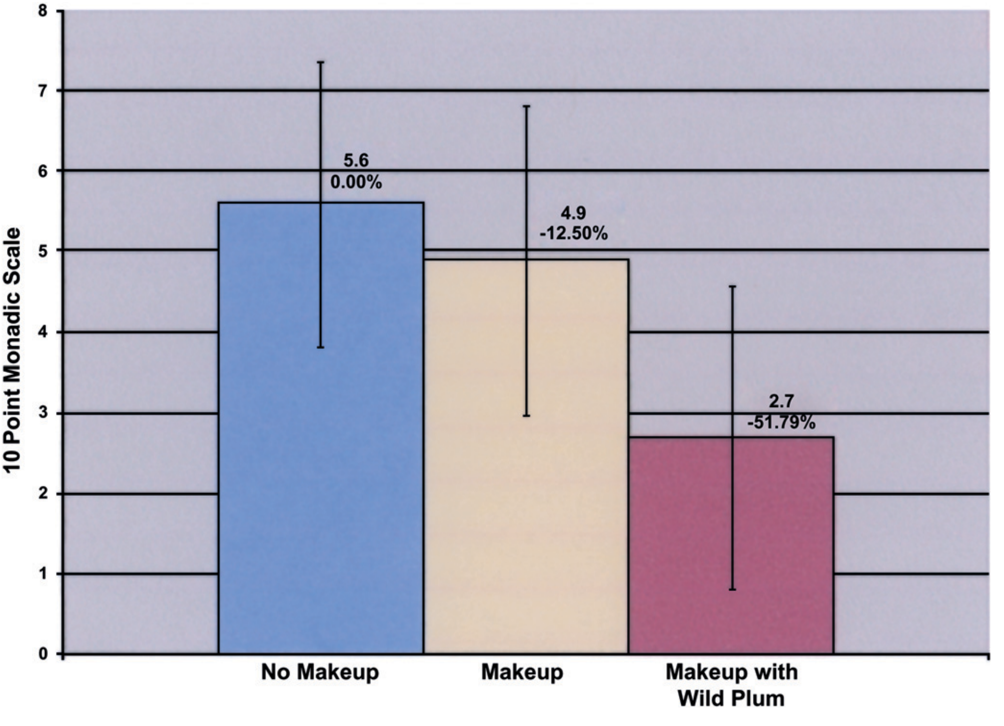


**Figure 4.** Results from an interview comparing images without makeup, with makeup, and with makeup containing Wild Plum using a ten-point monadic scale, with one [1] representing the fewest, least prominent wrinkles and ten [10] showing the maximum of wrinkles. n = 10. Error bars represent standard deviation.

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($p > 0.05$). However, comparisons between images without Wild Plum compared to images with Wild Plum were statistically significant ($p < 0.05$). In many cases, inclusion of Wild Plum made a very significant ($p < 0.001$) visual difference on the visual monadic scale.

The cosmetics industry uses nearly insoluble or encapsulated materials in order to protect color and luminosity (22,23). However, caking can become a problem with insoluble particles, and if encapsulation is used, sacrifice of color may occur. Hence, the need persists for cosmetic formulations that not only hide the appearance of wrinkles and lines, but also enhance the glow of youthful skin. Wild Plum is insoluble in water, oil, and other common solvents used in cosmetic formulations, but can be solubilized in detergents like SDS (sodium dodecyl sulfate) and nonionic surfactants like Triton-X100, allowing easy removal from skin by washing with soapy water. Caking does not occur with Wild Plum, as low concentrations are able to give the desired effect. This allows for optimal formulation conditions, as washing away or leaching of Wild Plum becomes irrelevant. Thus, the new photoluminescent Wild Plum is versatile in application and has been designed to eliminate dullness or the lack of luminosity often seen in aged and photodamaged skin (4).

## WILD PLUM VERSATILITY

The Wild Plum compound is photoluminescent, displaying a glow of intense blue color. In Figure 5, a gradient of blue fluorescence is apparent when Wild Plum is formulated into a commercial foundation at different concentrations.

Wild Plum was also co-mixed with light-scattering materials that have refractive indices close to that of skin at 1.5–1.6 for the "soft-focus effect" (17,18). Such materials include talcum powder, titanium dioxide, barium sulfate, and pearlescent pigments. Each powder can be used independently or combined to create unique shades specific to the desired application. The versatility of Wild Plum affords incorporation into a variety of cosmetic formulations without affecting the consistency, thickness, viscosity, or color of the formulation. Figure 5 also shows that Wild Plum can be used at concentrations as low as 0.02%, which shows that it can be used in existing formulations without significantly changing their properties after addition.

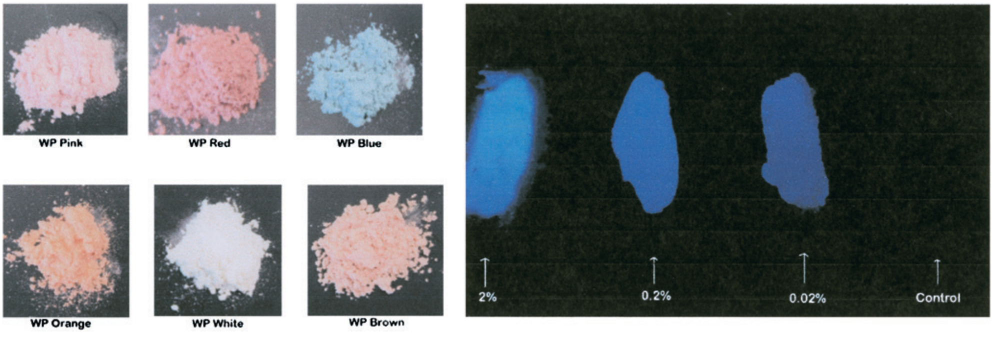


**Figure 5.** (Left) Wild Plum powders in different shades of color. (Right) Intense blue fluorescence of Wild Plum incorporated into commercial foundation at 2%, 0.2%, 0.02%, and the control; commercial foundation without Wild Plum (from left to right) excited at 365 nm with a UV lamp.

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WILD PLUM PARTICLE SIZE

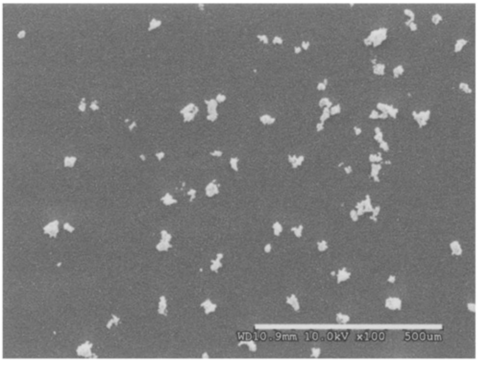
Particle size analysis data obtained by scanning electron microscopy showed that the amorphous particles ranged from 2.19 μm to 23.63 μm when mixed with talcum powder. (Figure 6). This makes Wild Plum ideal for toiletries, and avoids the range associated with toxicity and the biological effects of nanometer-sized particles (24,25). Furthermore, particle sizes between 2 μm and 20 μm provide good color properties and soft-focus effects (17). Depending on the intended use, the preferred average diameters will vary, based on desired granularity and texture. For example, a liquid facial cosmetic formulation comprising a fluorescent whitening agent as described above has a preferred particle size range of between 10 μm and 30 μm. A lipstick formulation containing Wild Plum should be formulated with particle sizes between 2 μm and 20 μm. Furthermore, admixing Wild Plum in a 2% concentration by ball-milling in micronized talcum powder does not cause aggregation to larger particle sizes.

WILD PLUM SYNTHESIS

The synthesis of Wild Plum is still proprietary. However, the compound was synthesized according to similar procedures published in United States patent application 11/863,475 (filed in 2007).

## WILD PLUM, FDA REGULATIONS, AND SAFETY CONSIDERATIONS

The FDA act exempts cosmetic ingredients from regulation except for sunscreens and color additives (26). A color additive is defined, essentially, as anything that imparts color. Wild Plum is considered a cosmetic ingredient that improves skin appearance, is not a color or dye, and thus does not require FDA certification. However, Wild Plum is an off-white powder and can be mixed with colored FD&C dyes to suit a particular cosmetic formulation. For example, as seen in Figure 5, Wild Plum Pink was prepared by mixing Wild Plum with FD&C Red No. 40 and FD&C Red No. 3. A blue shade was achieved by mixing Wild Plum with FD&C Blue No. 1 to specifically decrease redness and dark circles, enhance whiteness, and even out pigmentation, thereby increasing luminosity.



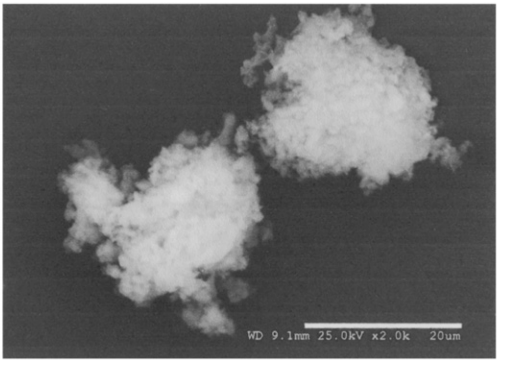




**Figure 6.** (Left) Scanning electron microscopic image of zoomed-out Wild Plum particles demonstrating an average particle size of 2–20 μm (bar = 500 μm). (Right) Image of individual Wild Plum particles (bar = 20 μm).

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The chemical composition of Wild Plum includes the fluorescent moiety 4,4′-diaminostilbene-2,2′-disulfonic acid. This component has been safely used in many cosmetic applications in the market, and is therefore not expected to be toxic (27). Preliminary skin irritancy studies have already indicated that there are no short-term adverse side effects, such as redness, swelling, or itching. More extensive skin irritancy studies are underway. Little or no skin irritancy is expected, given the facts that the concentration of Wild Plum in cosmetic formulations is low, at a level of 0.005% to 0.1%, and that the average particle is between 10 and 20 microns.

## WILD PLUM OUTLOOK

The leading edge of the baby boomer generation (born between 1946 and 1964) is a very important generation with purchasing power. Anti-aging products like Wild Plum offer this discerning and quickly growing demographic a solution to skin problems and imperfections without having to undergo painful and costly surgeries and peels; this also appeals to the growing male grooming market. The development of this new photoluminescent technology as an additive to existing cosmetic and toiletry formulations gives added value to the consumer.

## CONCLUSIONS

Wild Plum imparts optical brightness, color purity, and photoluminescence for use in applications such as cosmetic and toiletry formulations. The addition of Wild Plum to commercially available makeup indicates a dramatic improvement in skin appearance. Wild Plum leads to youthful-looking skin, as the appearance of wrinkles, pigmentations, and discoloration are diminished. Wild Plum powder can be incorporated into makeup products and is highly effective in covering skin troubles and creating a uniform, natural look. Preparations of Wild Plum could include any number of cosmetic and toiletry preparations, and work is under way to explore these possibilities. Future studies also include the development of Wild Plum particles that photoluminesce other colors such as green and red. Wild Plum is commercially available from Novel Chemical Solutions (Crete, NE).

## ACKNOWLEDGMENTS

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