Synthesis of new black pigment ; Carbon black pigment capsulated into the meso-pore of silica as black pigment in cosmetic

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1. Introduction

Recently the trend of new materials development is extensively and very actively progressing in the study of physical and chemical characteristics developing a totally new material along with the study field of recently discovered material modifying physically and chemically characteristics.

Among these fields of studies, one method to improve adaptation of inorganic material is the study of mesoporous materials. The most general way to synthesize mesoporous materials is to mold the very systematical mesopore into a corpuscle by using templates

These mesoporous materials are studied in a broad field such as catalyst, bio-sensor, optics, nano-composite materials (ceramic), polymer composite materials etc.

Among chemical compounds using mesoporous templates, surface active agent is used most generally, but surface active agent not only at a solvent is dispersed thermodynamically in a equal formation but also variety of template features differ at the active agent species. Surface active agents are divided by polar head group in to cation, anion, non-ionic, and amphiphiles etc. [1-8]

If higher density than CMC (Critical Micelle Concentration) of surface active agents is added into the solvent, rod micelles form inside the micelle. [9]

Mesoporous silica uses surface active agents as a template and is manufactured with sol-gel method resolving silicon-alkoxide with water.

Sol-gel reaction, as known as, divides into an early hydrolysis process, gel process by polymerization of sol that is hydrolyzed, and corpuscle's growth process.

At that point, polarity intermediate forming rod micelle reacts with surface active agents' polar head group and forms organic/inorganic hybrid material.

So in result, it decides the size and structure of the rod micelle and the pore. [10-14]

The size of the rod micelle is depended on the molecule shape and length of surface active agent's non-polar group.

Also, the structure of the pore is differed by the interaction of polar head group and silica polymer, interaction between surface active agent's non-polar group, and characteristics the solvent.

These interactions by surface active agent are showed by variety and concentration cylindrical form of hexagonal close packing structure or cubic form's structure. [15-16]

At this research, we synthesized organic/inorganic hybrid silica by using surface active agent as template at Lecithin, PEO from water and ethanol mixed solution

Even though, a large amount of Lecithin is used at the field of biochemistry and cosmetics, it has barely been synthesized using organic/ inorganic hybrid material.

Even though, carbon black is stable non-toxically, physically and chemically since the density is small and dispersion is bad it is hardly used as cosmetics.

At this experiment, surface active agent uses template synthesizes organic/inorganic hybrid silica and we used this template by heat treatment by heat treatment in deoxidization and synthesized black pigment making black carbon that was carbonized by pore trapped

These kinds of black pigments (called carbon-silica pigment from the following) can control the density by silica and carbon's relative quantity and can also control dispersion at hydrophilic solvent or hydrophobic solvent, so it can be used as very effective cosmetics. So at this research, we are trying to experiment the negligence of property of matter which put great effect on the use and safety of mascara.

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2. Experiment

2-1-1 Materials

Tetraethoxy silane (TEOS, 99.9%, Aldrich), Hydrogenated Lecithin(90%,), polyethylene glycole(3000Mw, 99.9%, Fluca), polyethylene oxide(PEO, 99%, Aldrich), ethanol(99%, Aldrich) were used as received, not more purified. High purity water was used as third-distilled, 18MΩ.

2-1-2 Preparation of silica with mesopore trapped carbon

The composition of the solution used for synthesizing the sample is listed on Table 1.

At this research, manufactured samples by altering Lecithin, PEO, PEG's density variably had shown excellent results with the composition on Table 1, therefore this thesis will be explaining samples synthesized with these composition.

Add an appropriate amount of Lecithin, PEO, poly ethylene glycol in 50 ml of alcohol, apply heat and dissolve.

After cooling this dissolvent at room temperature, add water and TEOS and for the dissolvent to be even reaction with diapering enough.

Finally, if you add ammonia water, heat it 50 degrees, the sol-gel reaction lasts for12 hours.

When reaction finished rinse 3 times with ethanol than filter,

Dry the white settlement at 110 degrees for 24 hours, than crush the dry sample by milling. There are two ways that dry samples are used. One is heat treatment in nitrogen gas atmosphere and another is heat treatment in a closed up melting pot.

Of heat treatment temperature such as 300, 350, 400, 450 degrees while synthesizing black mesoporous silica but the 400 degrees heat treatment sample has showed the best results, there fore in this thesis we will explain only about the results of heat treatment at 400 degrees.

2-1-3 Characterization

To confirm the synthesized organic/inorganic hybrid silicon's effective temperature during heat treatment we measured TG/DTA at temperature area of room temperature 700 degrees with 1 degree heating rate per minute among atmosphere.

You can confirm the pupil's shape and size formed by mesoporous silica by using the already widely known, Brag's equation '$2d\sin\Theta=n\lambda$'.

As the pore's diameter size is large, 2Θ's angle show smaller diffraction peak.

At the this research, we ensured the synthesized sample using 1° low angle, scanning rate of 0.1° per minute, and XRD of1-8° of 2Θ area.

The wavelength of X0ray source is 1.45 A by Cu K line.

The heat treated mesoporous silica particles of morphology is measured by using SEM.

sample	ethanol (mL)	Lecithin (g)	PEO (g)	polyethylene glycole(mL)	water (mL)	Amonia(mL)	TEOS (mL)
LPS	50	0.75	0.25	0	10	0.2	11.2
PES	50	0	0.75	0.25	10	0.2	11.2
LES	50	0.75	0	0.25	10	0.2	11.2

Table 1. Synthetic composition of mesoporous silica.

2-2-1 Mascara Materials

The basic formation and containment of mascara containing synthesized black pigments are organized on Table 2.

Ingredient		Content(%)
A	Di-Water	To 100
	Triethanolamine	1.0
	1.3 B.G	5.0
	Polyoxyethylene sorbitan monostearate	1.0
B	Carbon-silica pigment	7.0
C	Di-water	10.0
	Hydroxyethylcellulous	1.0
D	Methyl paraben	적량
	Propyl paraben	적량
	Carnauba Wax	3.0
	Stearic acid	3.0
	Cetyl alcohol	2.0
	Hydrogenated Stearyl Olive Esters	5.0
	Sorbitan stearate	1.0

	Microcrystalline wax	2.0
	Polyglyceryl-3 Bees wax	2.0
E	Acrylate copolymer emulsion	20.0
F	Di-Water	0.5
	Imidazolidinyl Urea	0.1

Table 2. Carbon-silica pigment containing mascara manufacture prescription

2-2-2 machinery

The necessary equipment to produce mascara as in oil paint equipment T.K Robomics(Tokushu Kika Kogyo,Co.,Japan) was used and as in dispersion Agi mixer (Hanyang, Korea) was used.

To predict mascara's visual transition, Sun rheometer(Sun Scientific co. Ltd., Japan) was used.

To measure blackness, colorymeter used Eye 3100 (Mecbeth Co,Ltd., U.S.A).

2-2-3 Manufacture

Mascara manufacture process using Carbon-silica pigment is made by using ingredients on Table 2.

First, diffuse phase C transparent by using Agi Mixer as transparent liquid state. Next, weigh phase A and heating at 70℃.

Mix the prepared phase A and C, heating at 80±2℃ than add to phase in and diffuse it well.

After diffusing phase A, B, C in homomixer at 2000~2200 for 5 minutes (temperature maintenance) add to phase put D in and homogenizing at 3000~3500 for 7 minutes.

While homogenized in cooling it at 53±2℃ add to phase E and phase F and homogenizing for 5 minutes.

Cool it till 30 degrees and complete manufacture.

Control was manufactured in a identical method.

At control, we used Iron Oxide black manufactured at Japan Totan Industry

3. Results and Discussion