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#### Original Research Paper

# Facile fabrication of light diffuser films based on hollow silica nanoparticles as fillers

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#### ABSTRACT

A light diffuser film based on hollow silica nanoparticles as fillers was facilely fabricated by coating a suspension of the particles in a UV-curable acrylate monomer solution on a cleaned glass substrate. The amount of the particles in the films was varied from 5 to 20 vol%. The optical properties and the light diffusing ability of the films were then studied and further compared to those of the cleaned glass. The result revealed that the films become opaque when the amount of the particles increases. The increment of the particles also leads to the formation of aggregated particles with a larger size and homogeneous dispersion as the FE-SEM images had been presented. Although the films exhibit the opacity, their total transmittance is still high and close to that of the cleaned glass transmittance. Moreover, the improved diffuse transmittance of the films is obtained from the increased amount of the particles. The increment of the particles also provides the different scattered light image sizes with a homogeneous light resulting in a distinct light diffusing ability. Those results indicated that the films based on the hollow silica nanoparticles as fillers are probably applied for light diffusing films in the LCD industry.

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#### 1. Introduction

Light diffuser films have both a high total and a diffuse transmittance since they are able to diffuse a point or line of light source; for instance, adjusting the angle of incident light [1,2]. Consequently, the light diffuser films have homogeneous light penetration. Therefore, light diffuser films have attracted a lot of interest during recent years for applying in the backlight systems of several liquid crystal display (LCD) devices [2-5]. At present, two types of the light diffuser films are widely achieved as good optical diffusion in the LCDs. The first type is called surface-relief diffuser films, which are mainly dependent on the microstructures of the surface layers [6–9]. Most of the microstructures are fabricated by complicated processes using expensive equipment [2,3]. Another type, namely volumetric diffuser films, is simply prepared by coating a mixture of filler and binder resin on optical material. According to the production of those two light diffuser film types, the study of volumetric diffusers has been conducted by many researchers. Moreover, the volumetric diffusers provide a uniform light scattering throughout the fillers, which play an important role on optical properties of the volumetric diffusers [1,4,5,10,12]. There are many kinds of fillers; for example, organic particles [1], inorganic particles [2,5,13,14] and organic/inorganic composite particles [3,4,15]. Principally, the filler-diffuser type films are related to the difference of refractive indices between the filler and the resin. When light passes through the interfaces of the filler and the resin, a refraction occurs as resulting in light scattering [2,4,5].

In particular, polymer-inorganic hybrid nanocomposite materials are considered as excellent option for multifunctional materials [16,17]. This can be attributed to the fact that they possess both polymers and inorganic nanoparticles. Moreover, such hybrid materials are capable of enhancing dispersion property, which is advantageous for mixing fillers and binder resins [18]. In addition, an aggregation of a small amount of inorganic particles is able to improve the bulk performance of polymer matrix for thermal, mechanical, and especially optical properties [2,5,13,14,19,20]. Those properties significantly affect the application of the fillerdiffuser films [2]. Meanwhile, the microspherical structure of fillers is widely exploited in optical diffuser films in the LCD industry [3,21,22]. However, using fillers which have a cubic structure in the fabrication of polymer-inorganic hybrid light diffuser films has not been reported in any involved research. Hollow silica nanoparticles comprise of a nano-sized hollow interior and a solid shell with the unique properties of low density, a high specific

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surface area, thermal insulation, optical properties; for instance, light-weight filler [1,5,23,24]. Due to these properties, the hollow silica nanoparticles have been much used as fillers light diffuser films. Their hollow structures can be expected to have a critical importance for light scattering as more light can penetrate into their hollow interior resulting in increasing light scattering [25].

In this study, the facile fabrication used to prepare a light diffuser film was a coating of mixed suspension of hollow silica nanoparticles and a UV-curable acrylate monomer solution by a bar coater. The UV-curable acrylate monomer has good mechanical and thermal properties; for instance, excellent optical properties [2,26–28]. The composition of the hollow silica nanoparticles was initially investigated in order to eradicate any unexpected result, which may affect the optical properties of the light diffuser films. The optical properties of the films containing different amount of the particles were then analyzed by a UV-visible spectrophotometer and compared to those of the cleaned glass in the visible region (400–800 nm). The quantitative effect of the hollow particles on the optical properties of the films was also investigated.

#### 2. Material and methods

#### 2.1. Hollow silica nanoparticles (HSiNPs)

The composition of HSiNPs was investigated using a thermogravimetric analysis (TGA, Thermo Plus TG-8120, Rigaku) measurement. The sample was placed in a platinum crucible for each analysis and then heated in the air to 800 °C at a heating rate of 10 °C/min. The TGA result was used to specify a certain temperature to eliminate some contamination. The morphologies of HSiNPs before and after calcination were observed using a field emission scanning electron microscope (FE-SEM, JSM-7600F, JEOL Ltd.) at an acceleration voltage of 15 kV with secondary electron image (SEI) and transmission electron diffraction (TED). The samples were dispersed in ethanol (EtOH, 99% Wako Pure Chemical Industries, Ltd.) by an ultrasonication. Then, the suspensions were dropped on a microgrid (STEM150Cu, Oken Shoji) and dried in the air. The density of the powder samples was further measured by a helium pycnometer (Ultrapycnometer 1000T, Quantachrome Instruments). The helium pycnometer was run in the multimode with a standard deviation of 0.05%. The samples were dried under vacuum at a temperature of 180 °C for 3 days in order to eradicate any error caused by any remaining air. The calcined HSiNPs, with the original raw material morphologies preserved, was used to prepare a light diffuser film.

#### 2.2. Facile fabrication of light diffuser films based on HSiNPs as fillers

An amount of the desired HSiNPs in the light diffuser films was studied as listed in the first column of Table 1.

The concentration of the suitable HSiNPs in the suspensions can be calculated using the equations on our previous study [26]. The calculated values are shown in the second column of Table 1. The known masses of the HSiNPs were dispersed in methyl isobutyl ketone (MIBK, 99.5% Wako Pure Chemical Industries, Ltd.) by the ultrasonication for 10 min. Meanwhile, UV-curable acrylate mono-

Table 1
Amount of the HSiNPs in the light diffuser films.

Amount of the HSiNPs in light diffuser films (vol%)	Amount of the HSiNPs in suspensions (vol%)
5	1.04
10	2.17
15	3.41
20	4.76

mer supplied by the JSR Corporation was diluted by MIBK to get 10 wt% UV-curable acrylate monomer solution. Each of the HSiNPs dispersions was combined with the 10 wt% monomer solution. Suspensions of the HSiNPs were obtained and then coated on cleaned glass substrates using a commercial bar coating machine (K101 Control Coater; RK PrintCoat Instruments, Ltd.) to obtain wet films. The cleaned glass substrates were obtained after cleaning in DI-water, EtOH, and acetone for 10 min in each solvent. The wet films were kept in the dark and then cured by a UV-photo surface processor (PL16-110D; SEN LIGHTS Corp.) for 5 min. This process aimed to induce photopolymerization. Consequently, the light diffuser films with different amounts of the HSiNPs as shown in the first column of Table 1 were obtained.

#### 2.3. Characterization of light diffuser films based on HSiNPs as fillers

The morphologies of the obtained films were observed with the FE-SEM. Before the FE-SEM observation, the films were directly dried on a hot plate at the temperature of 150 °C for 2 h since some residual solvent containing in the films may cause of unclear images. The dried samples were further coated by a thin layer of osmium tetroxide using an osmium plasma coater (OPC60A; Filagen). The films were also studied for their optical transmittance in the visible region (400-800 nm) using a UV-Vis-NIR spectrophotometer (UV3150; SHIMADZU) and compared the transmittance results of the films to those of a cleaned glass substrate. The light diffusing ability of all films was inferred by their scattered light image. The scattered light image was recorded by a digital camera performed on an apparatus together with a 532 nm laser (Z1M18B-F-532-PZ; Z-Laser). A film coated on cleaned glass substrate was placed on the apparatus in front of the laser lamp in about 15 cm length from each other. Subsequently, a scattered light image of the film exhibits on the dark background, which has the distances between them about 26 cm.

#### 3. Results and discussion

#### 3.1. Characterization of HSiNPs

#### 3.1.1. Composition of HSiNPs

The result of the composition of the raw HSiNPs was investigated using TGA and the result is shown in Fig. 1. It reveals two regions of weight loss. The first region of weight loss is observed in the range of 40–150 °C because of the removal of bound water (5%). Another region of weight loss about 25% (300–520 °C) can be ascribed to the dehydration of Si–OH on the silicates or the loss of some residual reagent such as surfactant, which is used to produce the HSiNPs [29,30]. Moreover, the incombustible residue



Fig. 1. TGA curve of the raw HSiNPs.

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