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Network Formation in Various Dispersing Medium of Hydrophobic Colloidal Silicon Dioxide

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Abstract. Colloidal silicon dioxide is a fine amorphous powder consisting of particles about 7-40 nm in size. This study utilized hydrophobic colloidal silicon dioxide, aerosil R 972, as gelling agent in non-aqueous systems. Aerosil R 972 could convert light mineral oil and Luvitol EHO® into gel at concentrations of 7 and 10% by weight, respectively. The appearance of the systems prepared from polyethylene glycol 400, 600 and light mineral oil was clear, but the others were turbid. There was no phase separation or color changes of the systems after temperature change of 6 cycles. The obtained gel exhibited very little change of pH and viscosity after stability testing. After aerosil R 972 was incorporated into the mixed media with different dielectric constant, all systems showed the decrease of a viscosity, especially, the formulations having low dielectric constant. The addition of aerosil R 972 into dispersing medium with higher dielectric constant provided higher viscosity of the system because of the mismatch of the polarity between particles and dispersing media. In case of the incorporation of aerosil R 972 into mixed media, the rank order of viscosity was PEG 400–glycerin> PEG 400–PG > PEG 400–water. All of PEG 400–glycerin mixtures were obviously converted into gel, while only PEG was not converted with aerosil R 972 addition.

Introduction

Colloidal silicon dioxide (colloidal silica or fumed silica or light anhydrous silicic acid or silicic anhydride) is a fine, white, odorless, tasteless, light and amorphous powder consisting of particles about 7-40 nm in size. Colloidal silicon dioxide is widely used in the oral and topical pharmaceutical products as binder and glidant in tablets and as suspending agent and a viscosity modifier in suspensions, ointments and suppositories. It is generally regarded as an essentially nontoxic and nonirritant excipient. However, intraperitoneal and subcutaneous injection may produce local tissue reactions and/or granulomas [1]. It should not be administered parenterally. The utilization of the hydrophobic colloidal silicon dioxide (aerosil R 972) as dry powder coating agent was previously reported which the drug release rate was decreased as the amount of aerosil R 972 increased [2]. The new oily gel base using colloidal silicon dioxide as gelling agent was previously reported [3]. The rheogram of this gel exhibited a thixotropic area that depended on the nature of oil and concentration of colloidal silicon dioxide incorporated. Aerosil R 972 could be applied as gelling agent in the hydrophilic/lypophilic microemulsion. This hydrophobic gelling agent contributed to improve a vehicle adhesion to the skin and increased the properties of the microemulsion as skin enhancer for the lipophilic drugs [4].

The aim of this study was to investigate the network formation behavior of hydrophobic colloidal silicon dioxide (aerosol R972) when it was dispersed in different solvents. The mechanism for network formation inducing the environmental viscosity enhancement from incorporated aerosol R972 was proposed.

Experimental

Materials

Aerosil R 972 control no. 1274041) were purchased from Wacker-Chemie GmBH, Germany. Isopropyl myristate, IPM (Ake-Trong Chemical 1985 Co., Ltd., Thailand) was employed as received. Glycerin, light mineral oil, polyethylene glycol 400 (PEG 400), polyethylene glycol 600 (PEG 600), propylene glycol, sorbitol and castor oil were supplied by P.C.Drug Center Co., Ltd., Thailand and used as received.

Methods

Preparation of system containing Aerosil R 972

The gels were prepared by dispersing Aerosil R972 at 2 - 10% by weight into hydrophilic dispersing media (water, PEG 400, PEG 600, PG, sorbitol and glycerin) and hydrophobic dispersing media (light mineral oil, castor oil, Luvitol EHO® and isopropyl myristate). The viscosity of all formulations was measured.

Evaluations

The viscosity of all prepared systems before and after incorporation with aerosil R972 was measured with Brookfield viscometer (Brookfield Engineering Laboratories,Inc.,USA.) (n=3). The pH measurement of hydrophilic dispersing media was conducted using pH meter (model 220, Corning, Germany). The gel clarity was also measured against a blank using UV-Visible spectrophotometer (Hitachi U-2000, Japan) at 480 nm. Phase separation or color change were visually observed for the prepared systems after the stability test with a temperature change of 6 cycles. For one cycle, all formulations were kept at 4°C for 48 h and then at 50°C for 48 h.

Results and Discussion

The clarity and homogeneity of miscible systems are shown in Table 1. Aerosil R 972 could not be dispersed into water, sorbitol, and glycerin because of the rather high polarity of these solvents. The appearance of the systems prepared from polyethylene glycol 400, 600 and light mineral oil was clear, but the others were turbid. There was no phase separation or color changes of the systems after the stability test with a temperature change of 6 cycles. All systems showed an increase in viscosity as the amount of aerosol R972 was increased. The apparent increase in viscosity was found when that amount of colloidal silicon dioxide could convert the liquid formulations into semisolid gels, especially, in the case of the system containing mineral oil (data not shown). The criteria to specify the gel formation in this study was that the systems could alter the characteristic from sol to gel. The 5 grams of systems was transferred into test tube of 1.5 cm diameter and the tubes were turned upside down. The systems must stay only in a test tube and did not flow down for at least 30 seconds. The guideline for this technique was applied from the determination of gel–sol transition state [5]. Aerosil R 972 could convert light mineral oil and Luvitol EHO® into gel at concentrations of 7 and 10% by weight, respectively. The viscosity of the gel systems was 13 – 2600 folds compared to the initial viscosity of the dispersing media (Table 2). There was the decrease of pH of the systems after incorporated with colloidal silicon dioxide (data not shown). The gel formulations exhibited very little change of pH and viscosity after stability testing. The viscosity of the mixed dispersing media having the same dielectric constant was different. In case of the incorporation of aerosil R 972 into mixed media, the rank order of viscosity was PEG 400–glycerin> PEG 400–PG > PEG 400–water (Fig. 1). All of PEG 400–glycerin mixtures were obviously converted into gel, while PEG only could not convert with aerosil R 972 into gel. The pH of the systems was lower than that of initial dispersing media because of the acid property of colloidal silicon dioxide [1]. The pH measurement of hydrophobic dispersing media was not investigated. Because pH is a measure of the concentration of hydrogen ion which should be measured only in aqueous solution since there are no suitable reference materials for calibrating a pH meter in non-aqueous systems or hydrophobic dispersing media.

Table 1 Physical appearance of the systems containing aerosol R972

Dispersing medium	Clarity Aerosil R 972	Homogeneity Aerosil R 972
Water	SP	SP
PEG 400	+4	+5
PEG 600	+4	+5
Propylene glycol	+1	+4
Sorbitol	SP	SP
Glycerin	SP	SP
Light mineral oil	+5	+5
Luvitol EHO®	+3	+4
Castor oil	+3	+5
Isopropyl myristate	+1	+4

* +1 to +5 = the rank order score of clarity and consistency from low to high
** SP = separated from dispersing media by floating

Table 2 Viscosity of the systems containing aerosil R972

Dispersing media	viscosity of the dispersing media(cps)	viscosity of the dispersing media after temperature change (cps)	Viscosity of the gel (cps)	Viscosity after temperature change (cps)
Mineral oil	45.67 ± 1.15	44.00 ± 0.00	118,733.33±18,094.57	149,466.67±601.01
Luvitol EHO®	27.00 ± 2.00	35.00 ± 0.00	560.00±120.00	640.00±105.83

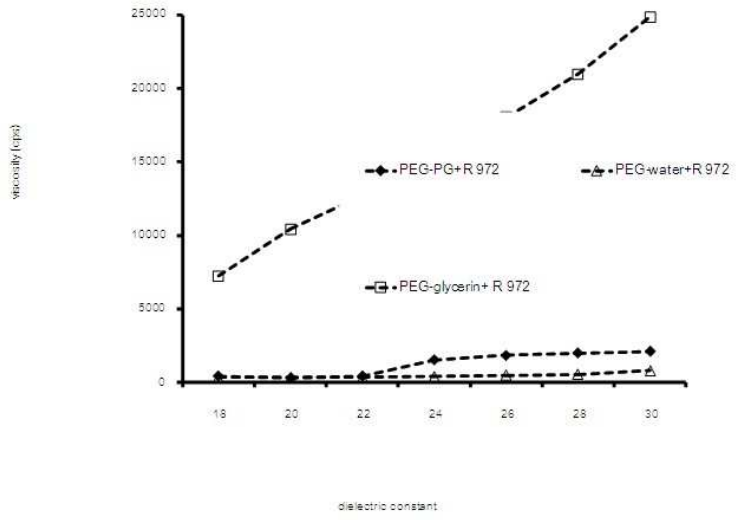

Fig. 1 Viscosity of various mixed media after incorporation with Aerosil R 972

A thickening effect was found after three-dimensional structure was developed [1,6]. At a suitable amount of colloidal silicon dioxide, the three-dimensional network led to the immobilization of a dispersing medium and the gel formation [1,6-8]. This phenomenon typically occurred when there was a mismatch between the chemical nature of the particle surface and that of the liquid. Aerosil R 972 could convert only light mineral oil and Luvitol EHO® into gels, while it could not convert high polar dispersing media i.e. water, sorbitol, glycerin and PG into gels. Aerosil

R 972 is the hydrophobic type of colloidal silicon dioxide. The silanol groups on the surface of Aerosil R 972 were chemically modified with dimethyldichlorosilane [6]. Therefore, it is hydrophobic and hard to be wet with high polar dispersing media i.e. water, sorbitol and glycerin. Generally, the nonpolar surfaces of aerosil R 972 might have a tendency to form connecting bridges in polar dispersing media, but the failure in gel formation was found in rather high polar dispersing media such as PG, which could be incorporated with aerosil R 972. Both nonpolar (dimethyldichloro) and polar (residual silanol) components on aerosil R 972 surface can exhibit either polar or nonpolar characteristics. Depending on the extent of mismatch between particle surface and liquid, the nonpolar dimethyldichloro groups would tend to cluster together in highly polar dispersing media [7]. On the other hand, the polar silanol would tend to associate in nonpolar dispersing media. In this study, light mineral oil and Luvitol EHO® might have some suitable properties which could be converted into gel with aerosil R 972. Owing to the obtained viscous environment, this system could be employed as the main of device for retardation or prolongation of active compounds liberation.

Conclusion

Network formation inducing the gel formation of aerosol R972 was evident when the dispersing medium was light mineral oil or Luvitol EHO®. PEG 400–glycerin was also the dispersing vehicle that aerosol R972 could convert into high viscous system. The obtained gelling systems could be applied as anhydrous drug delivery system for controlled the active compounds release.

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