

# Articles

## Unique Fluid Ensemble including Silicone Oil for the Application of Optical Liquid Lens

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The fluid ensemble in the liquid lens, which is composed of electrolyte and silicone oil, is the key material system to vary the focal length processing of the electrochemical desorption. In order to characterize the capability of the liquid lens according to response time and optical range, we prepared a fluid ensemble comprising the electrolyte and oil. To elucidate the physical mechanism of the effective response time, we examined the viscosity dependency while satisfying the requirements for the density and refractive index of the electrolyte and oil, respectively. The characterization results indicated that the response time (up and down) is influenced by the viscosity of the electrolyte and oil. On this basis, we prepared a fluid ensemble capable of reversibly adjusting for the focal length of the liquid lens, as well as the response time. The ensemble is applicable to various systems such as micro-lens and optical sensors.

**Key Words :** Auto-focus liquid lens, Electro-wetting, Aqueous electrolyte, Organic silicone oil, Refractive index

### Introduction

The liquid lens is attractive for a variety of applications, such as camera and optoelectronic devices,<sup>1,2</sup> as it incorporates the functions of auto-focus and zoom while satisfying the requirement for compact size, low electric power, and fast response time. The application of these abilities of liquid lens promotes price competitiveness because conventionally the functions would lead to camera modules too expensive for low-end applications like mobile telephones.

The physical principle of the liquid lens is based on electro-wetting by interfacial tension between the aqueous electrolyte and insulator coated on the electrode.<sup>3-5</sup> The contact angle of the aqueous electrolyte on insulating surfaces such as polymers or glass can be controlled by electro-wetting that is the modification of the electric charges at the solid-liquid interfaces.<sup>6</sup>

By applying electro-wetting to two-liquid systems with and without the voltage applied to electrodes, the curvature of the aqueous electrolyte-insulating oil interface alters from convex to concave that could act as a lens with the functions of focus and zoom for the image.

The fluid ensemble in the liquid lens consisting of aqueous electrolyte and silicone oils is the key material system capable of varying the focal length with the electrochemical desorption process. Much research has concentrated on the viability of two-liquid systems for lens within the frame-

work of electric devices.<sup>7</sup> However, there has been little investigation into the requirement of a liquid which satisfies the prior condition for optical device.<sup>8</sup> The acceptable condition of electric application is severe, so that a mobile telephone employing a liquid lens should be stabilized at  $-30$  to  $+70$  °C. The physical and chemical properties of the liquid depending on temperature have not been seriously considered.

In this paper, we prepared a unique optical liquid lens with variable focal length achieved by changing the contact angle between the aqueous electrolyte and organic silicone oil interfaces, reversibly, which is possible by changing the electric field and composition of the silicone oil components. To improve the electro-optic property and thermal reliability for the device, the sterically hindered siloxane structures were investigated as an insulator for the liquid lens since it may interrupt the intermolecular interaction between each other which results in low viscosity and good liquid solubility.

Furthermore, we investigated the relationship between the characteristics of the liquid ensemble and the response time for changing the focal length of the liquid lens which is applicable to various lens system such as micro-lenses and optical sensors.<sup>9</sup>

### Experimental Section

In the general procedure, 1,6-dibromohexane, 1,2-pro-

panediol, glycerol, silicone oils, and other chemicals were purchased from Aldrich and used without purification. Electrochemical potentials were controlled using a Priston Applied Research Model with 371 potentiostat. The response time for the liquid lens was measured as described in the literature.<sup>11</sup> The parylene layer was coated to a thickness of 5  $\mu\text{m}$ .

The electrolyte solutions were prepared with the following compositions. The  $\text{Na}_2\text{SO}_4$  aqueous solution ( $1 \times 10^{-2}$  M) included 1,2-propanediol and glycerol as an antifreeze in 65% (W/W). The LiCl solution included 10% or 20% (W/W) of LiCl, and *n*-propanol (0%, 30%, 40%). The chemical compositions were controlled to produce similar density and refractive index in the electrolytes.

The insulating oils were prepared with the following compositions: FE-1 (TST: 84%, 1,6-dibromohexane: 16%), FE-2 (TST: 82%, 1,6-dibromohexane: 18%), FE-3 (TST: 79%, 1,6-dibromohexane: 21%). The chemical compositions were controlled to achieve a similar refractive index in the insulating oils.

Regarding instrumentation, the response time of the liquid lens was measured as described in the literature.<sup>11</sup> Thus, the transparent liquid lens was prepared as shown in Figure 6. The beam of the laser diode was focused with the liquid lens, and a small pin photodiode, placed behind the liquid lens, was used to detect the beam intensity through the liquid lens. With voltage application, the beam intensity according to time was measured in the photodiode.

## Results and Discussion

**Structure of Liquid Lens System.** The liquid lens system consists of several components such as the aqueous electrolyte, insulating silicone oil, insulator, and electrode.<sup>10,11</sup> The principle of the liquid lens is based on electrostatic control of the interfacial tension at the aqueous electrolyte and insulator coated on the electrode. The hydrophobic insulator is coated on a plate electrode in order to control the surface wettability and decrease the contact angle of the hydrophobic silicone oil which can sustain the lens shape of the non-miscible liquid ensemble. Thus, a parylene or organic polymer layer could be used as an insulator.<sup>11</sup>

The non-miscible fluid system comprises two components: the electrolyte, which controls the contact angle, and the insulating oil that should have a higher refractive index. In addition, each liquid contains several components having a similar density to each other, so that the fluid system can

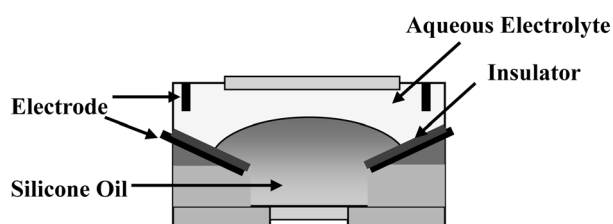


Figure 1. The cross-section of the surface of the liquid lens system.

properly act without any interference from gravity.

Figure 1 illustrates the cross-section of the surface of the liquid lens system containing two immiscible liquids with different refractive indices. An electrode directly contacts the aqueous electrolyte solution to apply the electric field between the conductive solution and the other electrode across the insulator.<sup>8</sup> By applying a voltage to the electrode, the contact angle of the aqueous electrolyte solution on the insulator decreases. Therefore, the shape of the organic silicone oil that has the higher refractive index changes simultaneously, which could be controlled to adjust the focal length with the applied voltage. The electrochemical behaviors of the on- and off-states are shown in Figure 2.

We tried to improve the liquid lens device characteristics to verify the materials of the electrolyte and insulating fluid. Thus, the two liquids in the ensemble should be formulated to have densities and viscosities as close as possible but a refractive index as different as possible.<sup>10</sup>

**Components of the Liquid Ensemble-Electrolyte.** When a voltage is applied to the electrode in the liquid lens, the water molecules tend to be attracted toward the insulator due to the electron-wetting properties, mainly due to increasing electrostatic interaction, as shown in Figure 3.

Concomitantly, the insulating oil with higher refractive index should change its shape to act as the fluid system for the variable-focal length lens. Thus, the liquid system, including aqueous electrolyte and silicone oil, is the key material in the liquid lens.

Usually, the electrolyte is required to be non-miscible with insulating oil, have a high conducting property, and be chemically stable. The  $\text{Na}_2\text{SO}_4$  aqueous solution ( $1 \times 10^{-2}$  M) has been used as the electrolyte for the electro-wetting experiment.<sup>6,11</sup> We initially checked the possibility of using  $\text{Na}_2\text{SO}_4$  aqueous solution as electrolyte, but failed, mainly due to the unstable property in the liquid system, as shown in

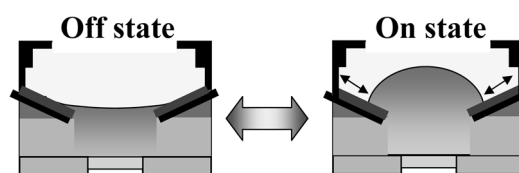


Figure 2. Electrochemical behavior of liquid ensemble in on- and off-state with and without the applied voltage.

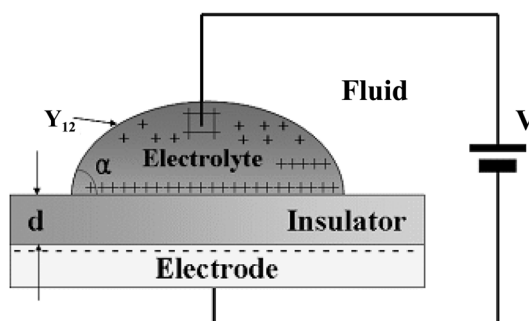
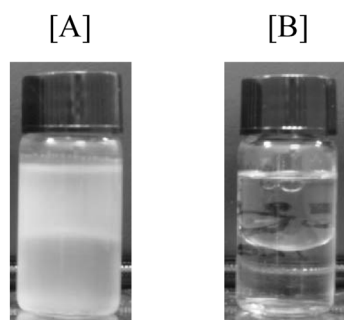


Figure 3. Electro-wetting properties in the liquid lens.



**Figure 4.** Liquid systems of  $\text{Na}_2\text{SO}_4$  [A] and  $\text{LiCl}$  [B] aqueous solutions after the thermal test at  $85^\circ\text{C}$  for 96 hr.

Figure 4.

The solution started to become muddy after the thermal test at high temperatures such as  $85^\circ\text{C}$ , as shown in Figure 4, [A]. Recently, we changed the electrolyte to  $\text{LiCl}$  aqueous solution due to its superior thermal stability, solubility, and conducting property compared to  $\text{Na}_2\text{SO}_4$  solution. It is important that the liquid lens should operate at a wide range of temperatures for portable applications such as mobile phone cameras. In our experiment, antifreeze was added in the aqueous electrolyte solution to depress the freezing point of the solution. Table 1 shows the difference of conducting property and refractive index for two solutions in which the chemical composition was controlled to have similar physical characters.

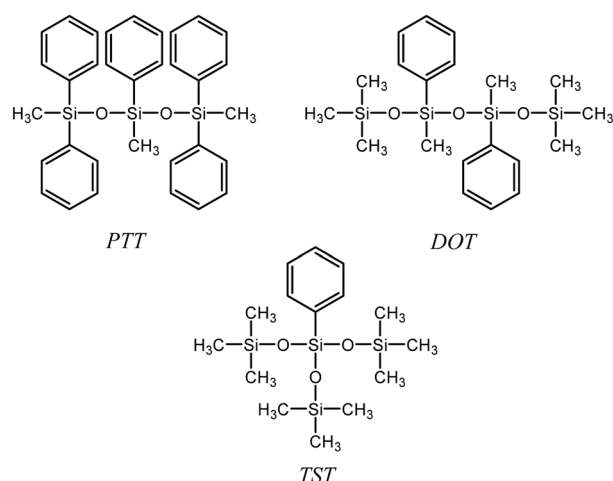
**Components of Liquid Ensemble-Silicone Oil.** The insulating oil should be non-miscible with the aqueous electrolyte, and a non-conductive material with a high refractive index, as noted above.

In our experiment, we selected silicone oil as the insulating oil, because of its non-conducting characteristics, thermal and chemical stability, and volume stability compared with other organic solvents, in addition to the ease with which its refractive index can be controlled. In particular, thermal expansion and shrinkage at different temperatures is one of the problems with insulating oil. The thermal expansion of silicone oil, at in 1.4-1.5 in our experiments, is very small compared with other organic solvents. We initially selected a silicone oil structure that has several phenyl moieties in the structure to control the refractive index and density of the liquid.

The structure is shown in Figure 5. Also, various amounts of 1,6-dibromohexane was added to control the density and viscosity of the solution (see experimental section). We

**Table 1.** Physical properties of the two electrolyte solutions

	$\text{Na}_2\text{SO}_4$	$\text{LiCl}$
Salt Concentration	0.2%	10%
Antifreeze Concentration	65%	40%
Density ( $\text{g}/\text{cm}^3$ )	1.087	1.092
Viscosity ( $\text{mP}\cdot\text{s}$ )	11.5	8.2
Refractive Index ( $n_{\text{D}}^{20}$ )	1.412	1.401
Conductivity ( $\text{mS}/\text{cm}$ )	0.228	22.8

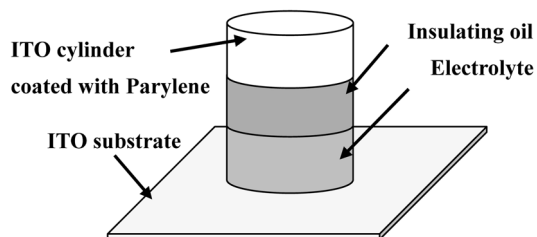


**Figure 5.** Silicone oil structures considered in the experiment.

believed that both liquids should have perfectly equal densities with spherical shape, independent of device orientation, and rather insensitive to external vibration. In the case of PTT and DOT which have several phenyls in siloxanes, as shown in Figure 5, the solution started to muddy after the thermal treatment at  $85^\circ\text{C}$  for 96 hours. As more phenyl moieties were induced, the solution became cloudier in the liquid ensemble. As mentioned earlier, the insulating oil should be formulated to have a refractive index as high as possible with consideration given for the power consumption and the response time. On the other hand, there is a trade-off as the high refractive index tended to make the solution hazy when it was dispersed into the aqueous electrolyte solution, in comparison with that of the low refractive index silicone oil.

We investigated many chemical structures of organo-siloxane to search for a solution to the above problem. Interestingly, the sterically hindered siloxane showed good physical characteristics and chemical stability, because the hindered types, such as TST in Figure 5, may interrupt the intermolecular interaction between siloxanes which results in low viscosity and good liquid solubility. In addition, the oxide group of silane is placed in the middle of the structure that can also be hindered from the thermal and chemical shock. In this experiment, we selected the TST structure as the insulating oil for the liquid lens system.

**Behavior of Liquid Lens by Electro-wetting.** In this section, we investigate the behavior of the liquid lens by electro-wetting. We have chosen a simple way to investigate

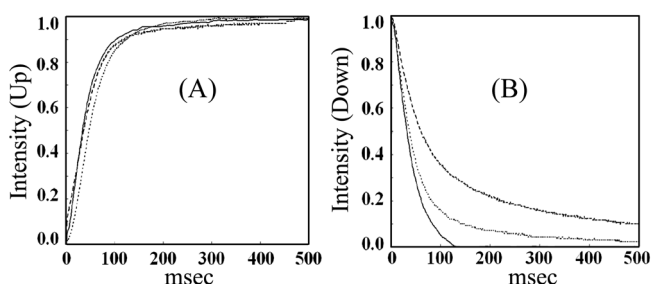


**Figure 6.** The liquid lens used in this experiment.

**Table 2.** Physical properties and measured response time of liquid lens

		Density (g/cm <sup>3</sup> )	Viscosity (mPa·s)	Refractive Index	Conductivity (mS/cm)	Response Time (mS)
FE-1	Electrolyte	1.083	6.1	1.389	40.8	53.4 <sup>a</sup> , 54.7 <sup>b</sup>
	Insulating	1.082	14.4	1.498	—	
FE-2	Electrolyte	1.092	8.2	1.401	22.8	43.2 <sup>a</sup> , 35.9 <sup>b</sup>
	Insulating	1.089	12.4	1.498	—	
FE-3	Electrolyte	1.104	2.8	1.372	— <sup>c</sup>	46.7 <sup>a</sup> , 102.9 <sup>b</sup>
	Insulating	1.104	11.9	1.498	—	

<sup>a</sup>Response time for voltage application. <sup>b</sup>Response time for shutting down the voltage. <sup>c</sup>Beyond the range.



**Figure 7.** Time response properties of various liquid lenses after applied voltage (A), and shut down the voltage (B). (small dash line: FE-1, straight line: FE-2, long dash line: FE-3) Silicone oil structures considered in the experiment.

the behavior of the liquid lens, as shown in Figure 6. Parylene-coated ITO cylinder was used as the liquid lens while the inside of the ITO cylinder was filled with liquid ensemble. The liquid orientation is arranged in the top of the silicone oil solution and in the bottom of the electrolyte salt solution. The ITO substrate and cylinder were used as the electrode and counter-electrode, respectively.

The response time of the liquid lens was measured as described in the literature.<sup>11</sup> Thus, the beam of the laser diode is focused by the liquid lens, and a small pin photodiode is placed behind the liquid lens. We controlled the refractive index of the silicone oil mixture by changing the concentration of the components to yield the correlation with viscosity and response time (Table 2).

While applying and shutting down the voltage, the beam intensity according to time was measured in the photodiode, as shown in Figure 7, and can be related to the focal length. Interestingly, FE-2 shows the fastest response after voltage application, despite having a higher electrolyte viscosity compared with that of FE-1 and FE-3 (Table 2). The measured response time of reconstruction after shutting down the voltage showed that FE-2 had the fastest reconstruction behavior compared with the others, as shown in Table 2. We attempted to increase the response time with changing viscosity of the electrolyte but failed. At excessively high viscosity, the response time dramatically increased. In addition, FE-3 exhibited the harmonic oscillating phenomena<sup>2,11</sup> after voltage application because the viscosity of the electrolyte liquid was too small.

We believe that the viscosity of the liquid is a key factor to control the response time and that they are also correlated

with each other strongly in the liquid ensemble, as shown in Table 2 which shows the physical properties and measurement of the response time of each liquid system used in the experiment.

We have to emphasize that the difference of viscosity between the electrolyte and the insulating oil is an important factor in the reconstruction time. In the case of FE-2, the difference was small compared with the other systems that could also reduce the, for FE-3, although the viscosity of the insulating oil was relatively small, the difference with that of the electrolyte solution was large and may have caused the slow response for the reconstruction.

The further optimization of the liquid ensemble, including synthesis of silicone oils, composition control, and construction of lens, is in progress.

## Conclusions

We prepared a unique optical liquid lens for which the focal lengths could be reversibly varied by changing the contact angle between the aqueous electrolyte and the organic silicone oil interfaces. By changing the electric field and composition of the silicone oil components, we could control the contact angle between the fluid system interface and the switching speed for reversibly adjusting the focal length of the liquid lens. In particular, the response time of the liquid lens was strongly correlated with the viscosity of the liquid ensemble, which is applicable to various lens systems such as micro-lens and optical sensors.

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