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## A squeegee coating apparatus for producing a liquid crystal based biotransducer

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Abstract. Cholesteryl ester liquid crystals were discovered with a new application in sensing traction forces of single cells. The liquid crystal bio-transducer is produced by manual scraping of liquid crystals onto the petri dish, in which the technique is highly subjective to the skill of the user to produce homogeneously spread liquid crystal substrates. This paper describes the development of an apparatus used to produce a liquid crystal substrate using squeegee coating technique. It consists of a biaxial mechatronic system which is synchronously controlled in vertical and horizontal directions scraping the liquid crystal substrates evenly on the surface of a petri dish. The thickness of the liquid crystal was profiled using laser diffraction technique and the homogeneity of the liquid crystal films produced was examined in a crossed-polarizing microscope. At an angular speed of 1500 rpm and under a shear stress of  $1.46 \pm 0.72$  kPa, the squeegee coating was found producing liquid crystal films at a thickness of  $132 \pm 23$  µm on the surface of petri dishes. With the application of this apparatus, evenly spread liquid crystal coatings with control thickness in petri dishes were consistently produced. This has overcome the major problem of manually coating the liquid crystal substrates using a cell scraper.

## Introduction

Liquid crystals (LC) were discovered with new applications in biosensing [1-4]. The immediate changes in the birefringence property of the nematic liquid crystals (LCs) in responding to enzymatic activities or reorganization of biological entity have enabled the detection of biological and biochemistry phenomenon without the need of labelling [1-4]. Recently, the shear sensitive cholesteryl ester liquid crystals were found with the potential in sensing traction forces of single cells [5]. This new biological relevant material with a Young's modulus of 80 - 100 kPa was found biocompatible with cells, stable at incubation temperatures (37 °C) and behaves linear viscoelastic below 10% of shear strain [6]. Cholesteryl ester liquid crystals are semi-solids and their physical appearance at cholesteric phase is alike to gel at room temperature [7]. The cell traction force sensing technique involves with quantification of discrete deformation lines in the LC surface induced by cells, in which the length of the deformation line is proportional to the force exerted by cells [5]. The formation of the deformation lines induced by cells in the LCs can be monitored via microscopy technique. With the use of LC based bio-transducers, traction forces of human keratinocytes (HaCaT) could be measured. In previous work [5], LC in isotropic phase was coated manually on a petri dish surface using a cell scraper and the thickness of the coating was controlled by a mask at a thickness of 100 µm. This technique is subjected to the skill of the user in order to produce evenly coated LC substrates.

In the manufacturing process of liquid crystal display (LCD), thin layer  $(1-2 \mu m)$  of LCs is spread on the polyimide coated indium titanium oxide (ITO) glass using rolling technique [8]. Electrostatic charge is applied to pre-align the orientation of the LC molecules sandwiched in the ITO cell [9]. The orientation of the LC molecules in the display device is switchable by the voltage applied to the ITO glass [10]. Dip-coating [11] is another technique proposed in coating LC cell but this coating technique required large amount of coating fluid and this seemed to be unsuitable for coating nematic or cholesteric liquid crystals that are usually quite costly at small volume. In this case, blade or slit coating might be a suitable technique for coating LC on a solid surface [12]. In considering the controlled thickness and consistency of the LC, squeegee coating technique might be a more applicable technique to coat LC in a petri dish. Therefore, an apparatus with squeegee coater customized for the coating of LC based cell traction force transducer was proposed for this work. The new apparatus is required to automate the coating process with an aim to achieve optimized LC coatings. In this paper, the development of the apparatus and characterization of the LC substrates produced will be reported and discussed.

## **Materials and Methods**

**Design of the Controller Circuit and Programming.** For this prototype, an electronic circuitry with an Arduino-uno board functions as the main controller was designed and fabricated (Figure 1a). As shown in Figure 1b, Arduino-uno was used to control the horizontal bipolar stepper motor which drives a linear slider that was fixed with a coating platform and the vertical stepper motor plays a role in lifting up and down the linear slider pre-fixed with a squeegee coater. The stepper motor used (Moatech, SPS-15RF) rotates at a step angle of 18° and a maximum slew speed of 1700 Hz. Both stepper motors were electrically driven by a dual H-bridge motor driver (L293D) with a maximum output current of 1 Ampere. The linear slider is constructed from a lead screw with a pitch length of 1 mm and 52 complete threads. Two limit switches were placed at both ends of the horizontal rails of the linear sliders. These switches prevent the sliders from moving out of the boundary electronically. A LCD was used to display the information relating to the number steps set and taken by both the vertical stepper motors.



Figure 1. (a) Schematic diagram of the squeegee coating apparatus, (b) flow chart of the controller circuit. A, F and LC denotes the contact area, force and liquid crystal.

The system was programmed using Arduino C language. The flow chart of the controller circuit is as shown in Figure 1b. Initially, the sliders are commanded to their pre-defined home position upon power-up or manually reset. Both the squeegee and the petri dish were driven synchronously by the stepper motor in each axis. The depth of the squeegee could be manually defined via a variable resistive dial. The stroke length of the horizontal axis could be adjusted via another variable resistive dial. Their corresponding stepping distances are displayed in the LCD. The squeegee coating process of the apparatus will be executed when a push button is activated. The coating process initiates when the squeegee coater moves down to the petri dish by the vertical linear slider controlled by a vertical stepper motor. Subsequently, the horizontal linear slider moves the motorized platform holding the petri dish (Figure 1a) to perform the coating mechanism. The coating process is completed when the squeegee head is lifted off the surface of the coating area. For measurement of the shear force induced by the linear motion produced by the motorized vertical linear slider, a newton-meter was used by attaching it to the coating platform. The force was monitored by varying the angular speed of the motor at 300, 600, 900, 1200 and 1500 rpm. Then,

the shear stress was calculated by the measured force (F) divided by the contact area (A) of the squeegee coater (Figure 1a). The force measurements were repeated three times.

**Preparation of Cholesteryl Ester Liquid Crystals.** Cholesteryl ester liquid crystals were synthesized and prepared as described in Soon at el. 2011 [6]. The physical mixtures of Cholesteryl ester liquid crystals in a vial were heated to isotropic phase at approximately 70 °C. LCs at the isotropic phase are ready for use in the coating experiment.

**Coating Process.** For the coating process, a petri dish will be placed on a platform of the coating apparatus (Figure 1a) where the petri dish holder is resided. Subsequently, approximately 20  $\mu$ l of cholesteryl ester liquid crystals melted at 60 °C in a vial will be deposited on the surface of the petri dish and the LC will be allowed to return to its cholesteric phase at room temperature before the coating process was initiated. Simultaneously, the bottom platform holding the petri dish moves horizontally in perpendicular to the squeegee therefore, achieving scraping of LC gel using the squeegee. After a few lateral movements, the blade is lifted up and the blade coating process ended. This should produce a film of LC with similar thickness coated on the surface of the petri dish for every repeat of experiment.

**Profiling the Thickness of the Liquid Crystal Substrate.** The actual LC substrates were coated directly onto the surface of the petri dish using the coating apparatus developed in this work. However, it was difficult to measure the thickness of LCs in the petri dish. Hence, LCs were coated on glass cover slips and this is achieved by offsetting the vertical linear slider with a distance of approximately 100  $\mu$ m off a glass coverslip placed in the petri dish. The thickness of the glass coverslip was determined using a micrometre. Then, the laser diffraction patterns of the cover slips with and without LC substrates were profiled using laser diffraction technique [13]. The images of the laser diffraction patterns for the glass coated with and without LC substrates were captured on a white board. When the thickness of the coverslips with and without LCs were obtained, the two measurements were subtracted from each other to yield the thickness of the LC substrates. The experiments for thickness measurement were repeated for 30 LC substrates.

**Determining roughness of the Liquid Crystals.** The roughness of the LCs at different angular speed of the motor was analyzed using microscopy technique. After the LC substrate is coated, liquid crystalline phases of the LC substrates were examined using an AxioPlan2 crossed-polarizing optical microscope (POM) at  $40 \times$  magnification. Images of the surface of the LC substrates were captured and the line profiles of LC surface were obtained using ImageJ software.

Results and discussion. Figure 2 shows a prototype of the squeegee coating apparatus which produces highly repeatable mechanism for coating. The apparatus for producing LC substrates comprising a platform having a petri dish and a stepper motor attached to the horizontal linear slider for moving the dish from a loading region to a coating region on the platform; and a coating assembly having a squeegee coater with a flat end which is in contact with the LCs and the coating member is configured to move upwardly and downwardly above the coating region by a second motor affixed to the vertical linear slider (Figure 2a-b). During the coating process, the flat end surface of the squeegee coater is drawn across the surface of the LC sample due to the forward and backward movements of the dish underneath the squeegee coater. This assembly with a squeegee tip (width  $\times$  length = 0.1  $\times$  1.5 cm) and the moving mechanism of the bottom dish created a shear force  $(0.12 \pm 0.02 \text{ N} \text{ at } 1500 \text{ rpm})$  to the coating material at a coating area of  $1.5 \times 3 \text{ cm}$ . The shear stresses were found decreasing linearly with an increase in the angular speed of the stepper motor (Figure 2c). At 1500 rpm, the shear stresses of the coating assembly is relative low down to  $1.46 \pm$ 0.72 kPa and the platform moved swiftly at this angular speed. Both motors at the highest angular speed producing the lowest shear stresses were found to exert minimum vibration and jerking at the coating platform. Figure 3a-b show the output of the film coated on glass substrates at angular speeds of 600 and 1200 rpm, respectively. The line profiles of Figure 3a-b as shown in Figure 3c indicate that the surface roughness of the LC coating is higher when produced under low angular speed. The crossed-polarized micrographs of the LC coated at an angular speed of 1200 rpm indicated even distribution of cholesteric phase as shown in Figure 3d. These results show that the



apparatus is able to produce LC substrates with high consistency at macroscopic and microscopic scales within 3 seconds of coating time.

Figure 2. Prototype of the squeegee coating apparatus with the (a) front and (b) rear views. (c) A graph of shear forces and force versus velocity of the stepper motor.



Figure 3. Photographs of the coatings produced at angular speeds of (a) 600 and (b) 1200 rpm, (c) profile of coatings, and (d) a micrograph of crossed-polarized LC coating produced at 1200 rpm. The blue lines indicate the lines taken to determine the line profiles of the coating based on the images. (Scale bar: 50 μm)

The line profile of the LC film produced at 600 rpm exhibited higher oscillations compared with the one produced at 1200 rpm as shown in Figure 3. Similar profiles of high oscillations were found for coating speeds at 300 and 900 rpm. The coating oscillations were greatly reduced at an angular speed of 1200 rpm (Figure 3c) but the smoothess coating was produced at 1500 rpm. At angular speeds much lower than 1500 rpm, the motor tends to exert high holding torque which impedes movement of linear sliders, therefore, leading to the production of uneven coating of CELC on the petri dish and glass surface (Figure 3a). In rheology, CELC was characterized by pseudo-plastic flow behaviour in which the complex viscosity of CELC exponential decreased with the shear rates (0.01 and 100 s<sup>-1</sup>) at room temperature [6]. The complex viscosity ( $\eta^*$ ) of the cholesteryl ester LCs at low shear rate (0.01s<sup>-1</sup>) is as high as 73.90 Pa·s in comparison with low viscosity at 0.2 Pa·s yielded at high shear rate (100 s<sup>-1</sup>) [6]. This indicates that when the shear rate of the linear slider is high, CELC would flow and the resistant for coating would be much reduced in comparison with the coating produced at lower shear rate. This explains the reason why the coating consistency is higher as compared when shearing the LCs at higher angular speed of motor.

After optimization of the coating speed of stepper motors and squeegee displacement from the surface of the petri dish, the squeegee coating apparatus was able to produce uniform film of LC at the desired thickness on the glass cover slips (Figure 3b, d). For the thirty samples, the thickness of the LC coating is approximately  $132 \pm 23 \mu m$ . The thickness of the LC coating produced is suitable to be used as a cell traction force bio-transducer [5].

**Conclusion.** A squeegee coating apparatus was proposed in this work to automate the process of producing LC based cell traction force bio-transducer. The system is able to produce suitable thickness of LC substrate in order to function as a bio-transducer. The coating mechanism is simple yet effective in spreading the substrate evenly onto the petri dish. However, the flatness of the squeegee coater, coating speeds, distance between coater and substrate, viscosity of the LC and the coating surface are important parameters that could influence the consistency and thickness of the LC substrate. Such apparatus works automatically and consistently.

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