

MICRODROPLET ELECTROWETTING ON FLEXIBLE PAPER-BASED MICROFLUIDIC CHIP

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ABSTRACT

This paper reports about the study of microdroplet reaction in electrowetting technique, which takes place in between two planar plates of electrode activated beneath the conductive droplet, with a low-cost material of production. In this paper, we show the result of frequencies effect to the droplet's velocity and contact angle by using the low-cost electrode material, which made from Aluminum sheet. The best frequency and voltage to be used with a 5 μ l droplet of 1400 μ S/cm KCl is 10Hz and 10Vpp respectively.

Keywords: Electrowetting; Microfluidic Chip; Microdroplet

INTRODUCTION

Microfluidics is a technology that can be used to manipulate microdroplets of liquid (for example from 10^{-9} to 10^{-18} liters) in terms of its motion and shape. Extensive studies on electrowetting are currently carried out throughout the world for various industrial technologies such as in point-of-care (POC) device [1], the study of liquids in Lab-on-a-chip (LoC) devices [2, 3], optical lens [4-6] and electronic display [7, 8]. Microfluidic systems have several advantages. The system can ensure faster results due to the simple fact that only small amount of reagent is required thus allowing a chemical process reaction time to be shortened drastically and reduce the wastage of reagent used. Electrowetting is one of microfluidic manipulation techniques. It involves voltage, frequency and electric field strength reaction to the physical properties of a droplet. It is a technique to wet a certain area using droplets that are mainly exposed to electric fields generated among conductive electrodes. It can be used in medical point-of-care devices [9].

In principal, an electrowetting works when the electric field lines penetrate into the ionic droplets and it induces polarization from two different ions and it leads to a movement [10]. When an electrical potential is applied to the conductive layer underneath a polarizable droplet, a layer of charge ions is built up at the interface between solid and liquid, which it reduced the interfacial energy between the liquid and

solid, γ_{SL} . There are several applications on droplet manipulation that can be done under the electrowetting technique such as transport [11, 12], mix [13, 14], and split the droplet [15]. Recently, Hongyao et al. have demonstrated a work of electrowetting that involved the creating, transporting, splitting, and merging of a droplet using electrowetting technique [16].

Electrowetting configuration is divided into two types; single plate and parallel plate. Figure 1 shows a generic structure of an electrowetting device for a single plate. The application of voltage as an actuation voltage, V_{act} between the electrodes causes θ to change because of the rate of ions in the electrode depends on the amount of voltage supply. The higher rate of ions attracted to the droplet ions, the stronger attraction it could make. When the contact angle is reduced, it will flatter respectively to the platform.

The parallel plates of the electrowetting consist of two electrodes; the upper plate and the bottom plate [17]. From Figure 2, the top-plate is used as the ground plate, while the bottom-plate as the control electrode where it controls the activated electrode. The voltage supply is connected to the ground plate and the control plate. Both electrode plates are insulated by the hydrophobic layer, while the dielectric layer coats the electrode array and the droplet is sandwiched at the center.

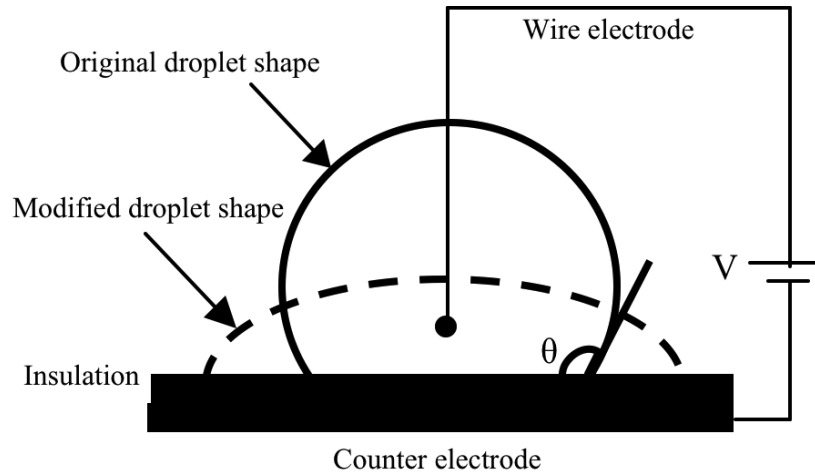


Figure 1: Electrowetting device structure for single plate [18]

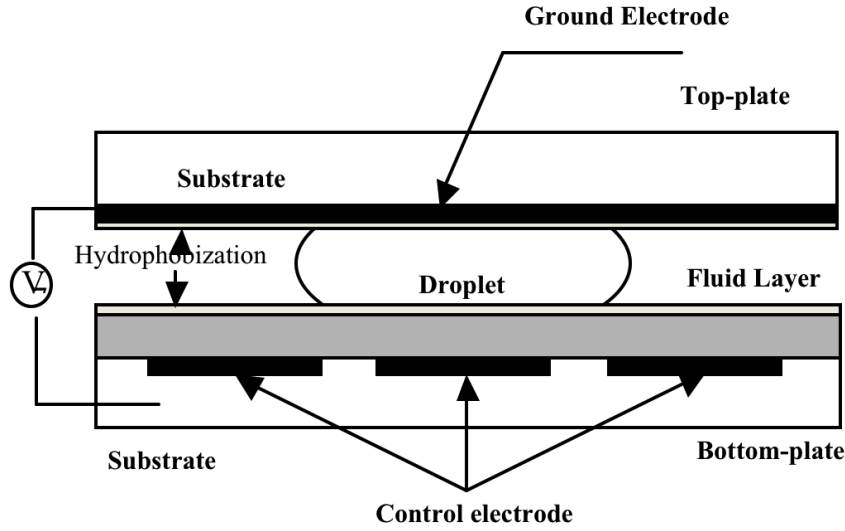


Figure 2: Electrowetting configuration for parallel plates [14]

Along the studies, attention to the reduction of the actuation voltage should not be neglected. Table 1 shows the actuation voltage obtained by several selected research works. All are due to different reagents and different metal-substrates junction used during the fabrication and experiment.

Table 1: Magnitude of Achieved Actuation Voltage

Reference	Actuation Voltage (V)	Reagent	Substrate
[1]	12.09V – 45V	DI Water	Chromium
[19]	7.8V – 17.64V	KCl	Chromium
[17]	25V - 35V	KCl	Chromium
[8]	80V - 140V	DI Water	Poly (3, 4-ethylenedioxythiophene) polystyrene sulfonate (PDOT:PSS) polymer
[21]	40V – 120V	Protein aqueous lysozyme and aqueous dithiothreitol	Carbon Nano-Tubes (CNTs)
[22]	30V – 120V	DI Water	Silicon

Voltage supply plays a big role in electrowetting operation. The manipulation of a voltage determined the contact angle reduction [24, 25], a velocity of droplet [18, 24] and the displacement of droplet travelled [9, 21], where all of these factors lead to the performance of electrowetting itself. In Lippmann's equation shown in Eq. 1 [26],

$$\gamma_{SL}(V) = \gamma_{SL}(0) - \frac{\epsilon}{2d} V^2 \quad (1)$$

where ϵ is the permittivity of the insulator, d is the thickness of the insulator, and V is the potential supply. The high voltage of electrode can contribute to the evaporation of the droplet where it will generate heat and can evaporate the droplet [17]. In addition, for example, in biomedical study, only at a certain level of high voltage, it can break down or damage a living cell because the cell is very sensitive to the environment i.e. when the droplet contains cells. Therefore, it is important to find out the best way to have electrowetting droplet manipulation in low actuation voltage to avoid harm to the cell.

Recently, a study on organic substrates has also been investigated where paper-based digital microfluidic devices are being developed [27]. This new considered material allows for a very cost-effective, biodegradable, recycle, biocompatible and good in flexibility [28, 29]. It has been reported in [30], a pair of electrode strips on paper was undergone electrowetting with a few range of low driving frequency. First, in the range of 1-10Hz, the droplet is moving following the electrode tracks. When it reached a range of 10-70Hz and 30-90Hz the motion was initiated by some perturbations. They found that within the range of 70-90Hz is the best range where it has strong unidirectional motion without external force is needed and under a low voltage of 70Vrms. Based on this finding, hypothesis was made where the lower frequency will be worked on and could enhance the droplet flow on the electrode track.

In the leading technology, many researchers are eager to figure out on more challenging topic when the electrowetting is executed on the flexible substrate [31]. Paper is the most promising and the best flexible material because it is portable (light-weight and flexible). It also low in cost and can come in any shape and size where all those make it easier to be designed as the platform. The flexible electrode is a crucial material that must be confirmed to have an efficient flexible device. There are flexible electrodes that have been tested with electrowetting such as graphene [32], copper [33], and indium tin oxide (ITO) [34].

Reported in [35], the combination of bare Aluminum (Al) and anodic electrolyte would actuate a droplet under a low voltage of 10V. Al electrode is known as the low-cost material and it has successfully demonstrated for electrowetting without electrolysis [36]. Both of these works have inspired the design of our microfluidic transportation chip as the low-cost, disposable droplet transportation strip. In this paper, we present the study of the designed chip is dependent on frequency under low voltage.

EXPERIMENTAL

Preparation of Microfluidic Chip

In this study, we focus on a single-plate of electrowetting. This is to observe the frequency effects on the electrowetting technique on one side of the flexible paper-based microfluidic electrowetting chip. The platform of the chip is the substrate, which is made by few layers. The platform layers are shown in step-by-step in Figure 3, while Figure 4 shows the electrode geometry from the top view. There are four steps needed

for this configuration.

The first layer is made from a piece of cellulose paper (Double-A paper), which is cut into small-sized of paper in size of 1.5 cm x 6 cm (width x length). This paper based substrate has a very thin thickness of 106.5 μm made up from Elemental Chlorine Free (ECF) and low bleach cellulose. It has the brightness of 102-104% and above. The color is white with whiteness measured graded in International Commission on Illumination of CIE167. Its roughness is 140 ml/min and the capacity is 95%. It is also inexpensive manufacturing of platform and easy to be folded, which is high flexibility.

Secondly, the cellulose paper will be covered by a thin layer of transparent adhesive film tape (Scotch®Tape 600) of 58 μm thickness. The transparent adhesive sheet is made from a cellulose film. The cellulose film has low permeability to liquid, air, oil, bacteria and common to be used as a packaging material. Due to the characteristic of permeability, the thin and transparent adhesive sheet is chosen as the protective layer to the cellulose paper to prevent any absorption of the liquid droplet.

Lastly, the Al sheet (Diamond) is patterned with determined size and shape and adhered on the top of an adhesive-unwoven sheet by using a pressure-sensitive technique that has been coated with an acrylic adhesive on both sides (Nitto No.5000ND). The thickness of the Al thin film and the adhesive sheet is about 16 μm and 320 μm respectively. In this paper, the dimension Al sheet as the electrode is 1.5 cm x 0.1 cm of length and width respectively and the gap between the two Al electrodes is 1 mm. Al has been used as the electrode in the flexible display device [7] and has been tested that the resistance of Al is very low, which in between 0.2 Ω until 0.5 Ω . Al is a very lightweight, soft and low strength metal that made it easy to forged, machined, cut, formed and welded. Thus, Al is easy to cut into small pieces due to the softness but strength. It also has high resistance to corrosion, a good conductor and can retain good ductility at subzero temperatures.

The fabrication is easy to handle because it does not involve with any high power equipment and can be prepared anywhere as long as it is constructed in a dry and clean area. Despite, the platform preparation has its own challenges; due to the manually handled by hand, the cutting process is very sensitive because of the small size in the range of millimeter down to micrometer has to be maintained in order to have similar size of the platform under duplication and repeatability process. Thus, during the running of the experiment, the safety (sharp scalpel) and cleanliness precaution must be taken care of especially on micron-sized dust, which may affect the droplet particle. When these are taken care of, the number of device duplication can be reduced with more experiments can be performed on one device.

Reagent and Dielectric of Microdroplet

As to reduce the force between droplet and electrode, a dielectric layer or film from oil lubricant is positioned on top of the electrode with a thickness of approximately 400nm. A layer of cooking oil (Naturel Sunflower Oil) has been used as the lubricant oil in this

experiment. One of the advantages of placing the dielectric film is, it can prevent the evaporation of the droplet [26].

A droplet of Potassium Chloride, KCl has been pipetted on the electrodes, in a volume of 5 μ l (5 μ l is selected by taking into account of the previous work in [37]) and formed a half-spherical shape. The volume of the droplet depends on the size of the electrode. In this experiment, the volume and placing of the droplet are made slightly larger than electrode size so that the droplet easily overlaps the neighboring electrodes.

The KCl is chosen as the tested droplet because it is the type of conductive liquid that has the stability of carrier charge, which can react at the same rate of response of proton and electron [38]. Besides that KCl also working as a transportation agent for the live cell such as bacteria and it is needed in biomedical technology in order to analyze and test the biological sample [39]. A fresh droplet of KCl is used for each experimental test to remove the evaporation effect.

The conductivity of KCl is measured by using Primo 2 Conductivity Tester (Hanna Instruments). The contact angle, C.A. was repeated more than 10 times on the working chip to have the consistency of result. However, 4 chips have been replaced to fulfill the consistency of result because one single chip only working good in less than three times.

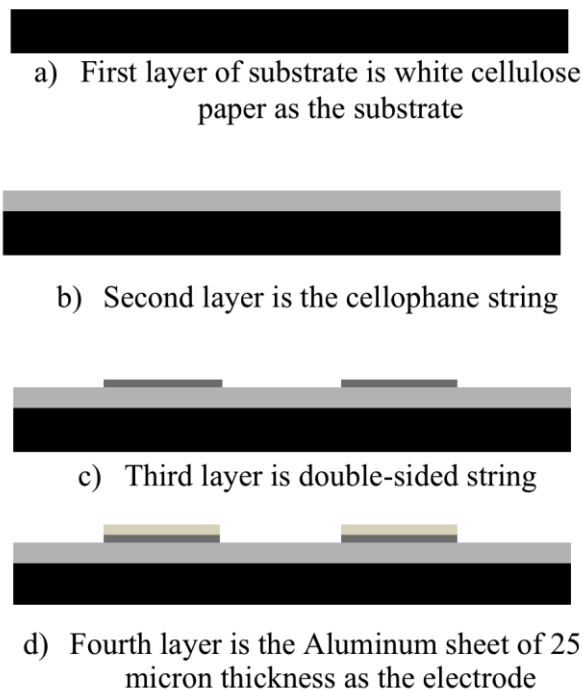


Figure 3(a-d): The step-by-step layer of chip fabrication

RESULTS AND DISCUSSION

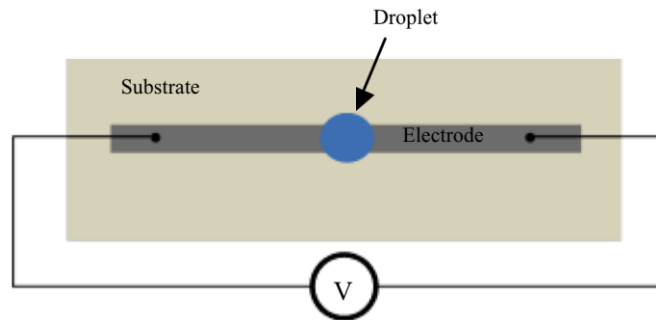


Figure 4: Electrode geometry for the transport of water droplets based on self-propelling mechanism from top view

Observation of the Droplet Motion with a Microscope

Originally the diameter of 5 μl KCl is about 4,000 μm . When AC voltage is turned on, the droplet slightly flattened while it heading towards the end of the path until at certain time it stopped moving. The movement at the edge of a droplet is like a worm movement. Same like reported in [40], where low frequency at 1 Hz has induced a droplet ratchet-like motion. The large deformation is observed in this experiment, where the shape of droplet changed due to the attraction charge of polarization at the droplet edge.

In this experiment, we observe that the droplets start moving along the electrode path when the electric potential is turn on. The initial direction of the motion is unpredictable, but once started, it flows persists until the droplet reaches the last point, which also unpredictable. Somehow, the movement will stop when the power supply is turned off. At $V=0$ V, the shape of the droplet is as shown in Figure 5a), the droplet is asymmetrically positioned using pipette at the middle of the electrode pair. Once the electrode is on, there will be one side of droplet edge act as leading edge and one side act as trailing edge. Like shown in Figure 5b), the leading edge is on the right-side electrode and the trailing edge is on the left-side electrode. This trailed phenomenon is the same as reported in [30].

The droplet actuation is observed using BX51 Olympus Microscope. The objective lens of microscope used is the smallest one, which is 5X while the eyepiece is 10X and total angular magnification of microscope image is 50X. In this experiment, the highest voltage that has been tested is up to 20 Vpp, and it does speed up the droplet motion. However, 20 Vpp is not a suitable rate of voltage where the heating is too much for the direct charging and end up with 10 Vpp as ideal voltage.

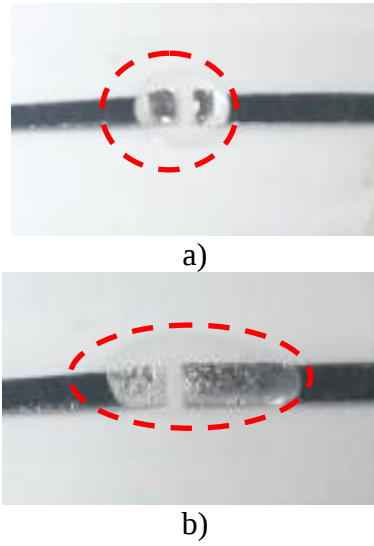


Figure 5: a) Droplet at $V=0$ Vpp, b) Droplet at $V=10$ Vpp

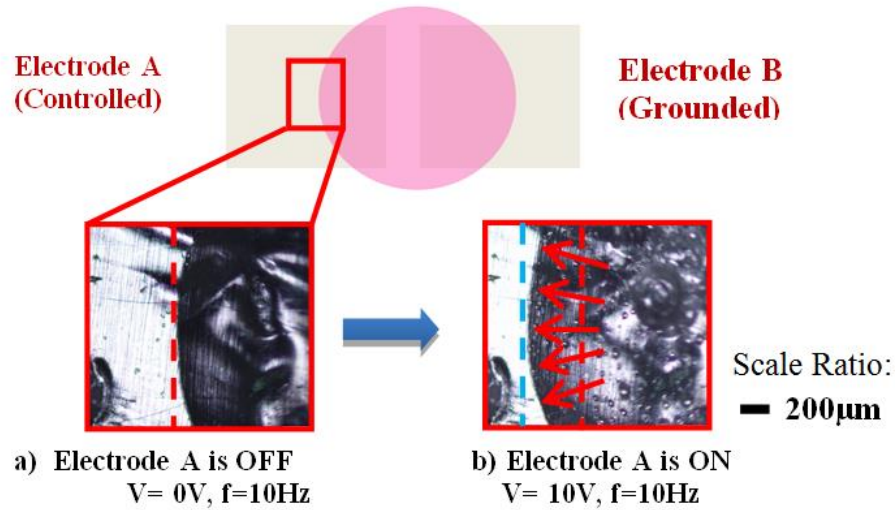


Figure 6: Edge of droplet taken under microscope observation, a) The voltage is OFF. b) The voltage is ON

Figure 5 is taken during the experiment is running with 10 Vpp and 10 Hz of voltage and frequency respectively. This is the best frequency that can be used for Aluminum electrode. The dashed red lines in Figure 5a) and 5b) show the droplet from the top view, before and after the electrode is activated respectively. While the dashed red and blue lines in Figure 6a) and 6b) show the final position of the edge of the droplet before and after the electrode is activated respectively.

Note that, the volume of the droplet is set with a suitable dimension of the electrode strip. Moreover, the conductivity can be increased or decreased by adding or reducing

the amount of KCl respectively in the solution. The result might give a different specification of frequency due to the changes in conductivity.

Contact Angle Reduction

The contact angle, θ (C.A.) reduction is one of the important characterizations in the electrowetting study. It is to show the movement of the droplet and the performance of wetting area. At this point, C.A. measurement becomes one major objective to maximize the range of contact angle. The measurement of θ is the angle measurement from the surface of solid-liquid phase to the surface of liquid-air phase [41]. The larger the wetting area along the electrode track, the higher reduction of the contact angle between the surface of the droplet and the substrate surface. Thus C.A. is measured using software image processing Stream Essentials from Olympus. From Figure 5 above, the droplet image is taken using the digital camera of 16 Megapixel from side view during the droplet is under experiment. The wetting area is larger under the microscope observation as shown in Figure 6b).

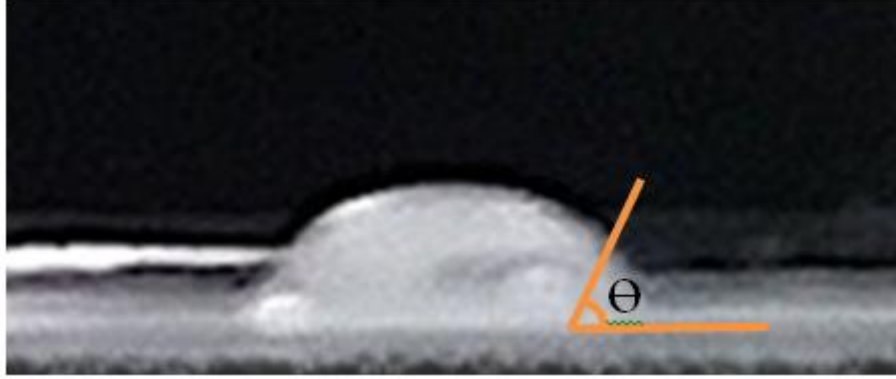
In order to ensure the platform is on a flat surface and steady condition, the bottom paper platform is adhered with tape on the microscope to avoid any small movement during the experiment. The droplet in Figure 7a) is the original shape after pipetted on top of the electrode, and Figure 7b) shows the flattened droplet after a few second the electrode is activated.

Based on Figure 8, the comparison has been made between KCl and DI water; it is obviously shown that DI water did not give any change of the contact angle, C.A. The y-axis on the figure is the result of reduction of contact angle where it is the subtraction between the contact angle before and after the applied voltage. For KCl solution, the frequency 10Hz has the most stable reading in term of consistency in repetition experiment of 5 μ l of droplet volume and conductivity of 1400 μ S/cm. It always becomes the best frequency that gives the highest reduction in C.A. This experiment was conducted within low frequencies because high AC frequency disregards the droplet conductivity and leads to disturbing the insulating droplet behavioral where it is discussed [42]. Besides that, at a very high frequency up to kilo and Mega Hertz, the droplet behaves like saturated object due to a too fast response of electric charges at the edge of the droplet.

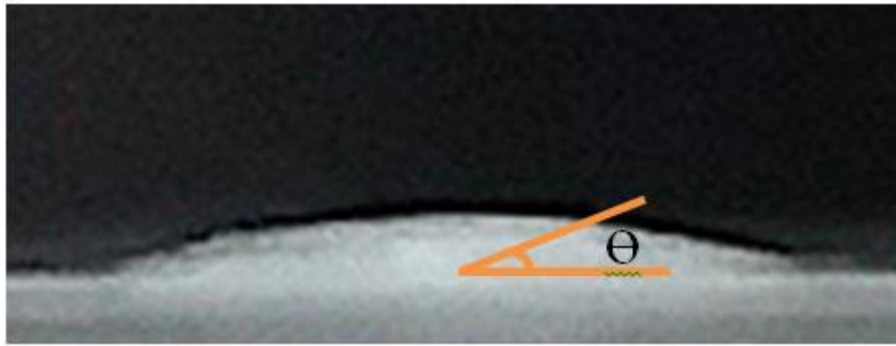
When the electrode is supplied with low frequency, the rate of charge accumulation is in stable mode and able to make an attraction very well. The C.A. saturation also becomes independent for high range (1k – 1kHz) of AC frequency [43].

When the external voltage supply connected to the left and right of the electrode, there will be a redistribution of charges and dipoles. This effect leads to modifying of surface energy at the interfaces as proven in Lippman's equation [44]. The presence of an accumulation of net charge at the interface reduces the surface tension due to the repulsion between two layers of net charge, as shown in Figure 9. The repulsion makes the droplet looks like moving forward and get the wetting area larger. In the figure, the

black line denotes as a layer of lubricant oil, which act as a dielectric medium between electrolyte and electrode.



a) Electrode is turned off.



b) Electrode is turned on.

Figure 7 (a-b): Contact angles of droplet are taken from side-view

$$f_c = \frac{G_l}{2\pi(C_d/2 + C_l)} \quad (2)$$

From Figure 9, the electric circuit has been modelled as in Figure 10 and critical frequency, f_c has been derived as in Eq. 2 [45]. G_l , C_l and C_d represent for liquid conductance, liquid capacitance, and dielectric capacitance respectively. Jones reported [45] that f_c is the maximum point of frequency where the droplet able to react to the voltage supplied. If the frequency used is above of f_c , the droplet is disabled from moving neither decrease contact angle. As calculated for f_c , the result gives 30 kHz. Experimentally, the f_c acts at 1 kHz because the droplet did not give any result at 1 kHz and above. However, the result only recorded until 100 Hz because above 100 Hz, the changes were very small.

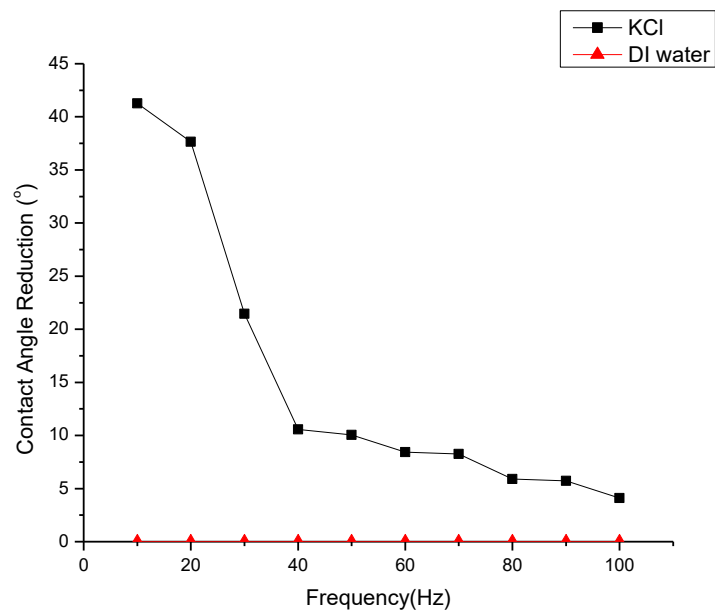


Figure 8: Graph of contact angle of droplet (°) versus frequency (Hz)

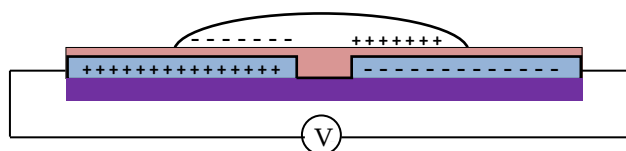


Figure 9: Charges distribution at the electrode-electrolyte interface

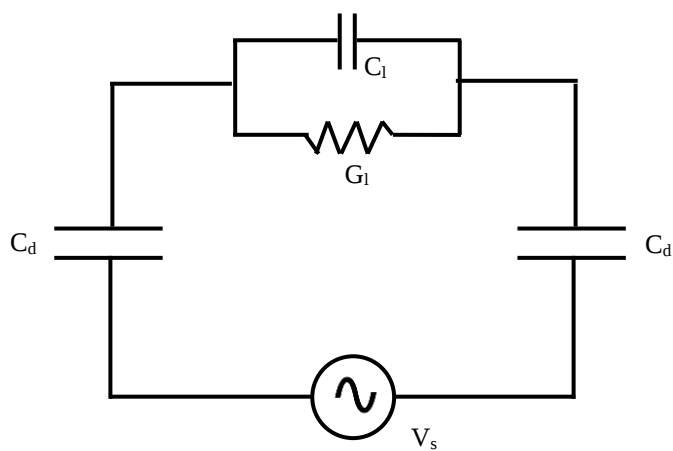


Figure 10: Electrical circuit model based on EW platform

Velocity Responses

When there is a motion, there will be a velocity. The velocity can be measured by the rate of distance between two points to the traveling time. The simple equation of velocity is as shown in Eq. 3;

$$v = x/t \quad (3)$$

where v , x and t denotes as velocity, the displacement between two points and time travelled respectively. The velocity of the droplet has been measured from its starting point when the droplet is at a stable, original or starts position and at the final position after it has been actuated by the activated electrode. The result for the velocity within two distance points with different frequencies is plotted as shown in Figure 11.

In this operation, the conductive droplet is assumed to directly touch the electrode. This condition is similar to a conductive solid touch an electrolyte and the electrical reaction between them is called electrical double layer (EDL) [46]. The conductive droplet on top of the electrode originally has static negative and positive charges, but then it is distracted and attracted to their opponent charge from the electrode itself and undergoes polarization process. There are two layers of opposite polarity ions as those positioned at the boundary of droplet and electrode when the voltage is applied. Thus, the polarization plays the role where the attraction of droplet ions and electrode ions leads to accumulation of ions, and the density of accumulation is strongest at the edge of the droplet. The attraction of accumulation actuated the droplet naturally and this reaction is called electrowetting, where the fluid initiated to wet the surface area of the electrode track. The worm-inched movement is observed earlier is believed due to the cycle of the AC pulse which always switching between negative and positive sign wave. The droplet motion reacted based on those pulse sign. However, after several pulse steps, the flow of droplets does not in worm-inched condition and it flows smoothly like no resistance or changes of pulse occur.

The graph concludes that the frequency of charge does affect the droplet motion, where higher frequency would slow down the speed motion until it begun to have a uniform speed [18]. The peak frequency of 40Hz shown in Figure 11 has the fastest speed. The graph is a representative of the experimental result with 5% of error during the measurement of distance. This error might come from the slippery effect of the lubricant oil. It also shows a comparison between two samples; KCl and DI water and only KCl solution give a motion result. It might be because DI water is not a conductive liquid and it cannot react well with the electrical effect.

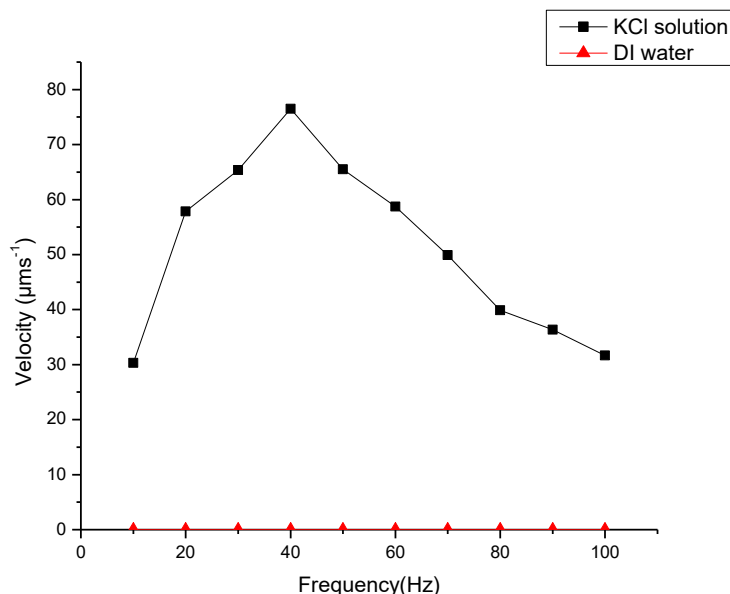


Figure 11: Graph of velocity for droplet motion within two distance points

Regarding Figure 8 and 11, both C.A and velocity respectively depend on the frequency. The aim is also to have a good electrowetting where the C.A does not reduce critically. Therefore, at 10 Hz, it is shown that the most reduction of C.A. happened when the droplet is flowing with steady velocity at 35 $\mu\text{m/s}$ and it is anticipated as the highest velocity of the droplet can flow.

In term of resilience and testability, the platform chip can be used maximum 3 times repetition with the same experiment. This has made the chip as a flexible and disposable microfluidic electrowetting chip. After three times, it will be disposed and repeated with a new one. This is because when the layer of the platform is exposed with too much wetting in such extensive time, the adhesive layer under the Al sheet will be swelled due to too much liquid immersion from the droplet and oil itself. The preparation of this paper-based microfluidic chip platform is easy and simple despite in this prototype it is handled manually by hand.

CONCLUSION

The electrowetting paper-based platform using the low-cost Aluminum thin sheet was capable of having a droplet manipulation under the voltage and frequency actuations. The Al becomes a track of the droplet to move, and it is ready to be improved for future device generations. Future work, we plan to use the present work as a guide. In conclusion, at 1400 $\mu\text{S/m}$ of droplet conductivity, with 10 Vpp and 10 Hz of voltage and frequency supply respectively, these are the ideal specification to have a low voltage and low-frequency electrowetting platform with a very low-cost electrowetting chip production. Using this low-frequency system, it follows the hypothesis mentioned

in the introduction where low frequency could enhance the flow of droplet on the electrode track.

This work is important to have a convenient and low-cost production of the microfluidic electrowetting devices. Further study on the effect of the voltage supply is ongoing for this topic.

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