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## **IMPROVEMENT OF MIXING FLUIDS EFFICIENCY IN LAB-ON-A-CHIP DEVICES USING ELECTROACTIVE POLYMERS AS ACTUATOR SYSTEM**

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### **KEYWORDS**

Lab-on-a-chip, mixing, acoustic streaming, heating, electroactive polymer.

### **ABSTRACT**

Rapid driving and mixing of microfluidic species are very important for several biomedical applications. However, these tasks are challenging for lab-on-a-chip systems due to the small channel dimensions involved. This paper presents the validation of an acoustic streaming system, based on a piezoelectric  $\beta$ -PVDF polymer, for improving the mixing efficiency in lab-on-a-chip devices that overcomes the microscale limitations. Preliminary experimental results proving the feasibility of that method are presented, as well as the processing conditions and optimization of the piezoelectric film.

### **INTRODUCTION**

Microfluidic involve the manipulation, control and analysis of fluids inside channels with dimensions in the micro- and/or nanorange and has become an important tool for chemical and biochemical analysis. The interest in this technology is based on the inherent performance gains that include: reduced sample size; higher degree of integration; enhanced potential for automation and control of submicroliter volume fluids; shortened response time; potential for improved analytical performance; reduced chemicals storage; safety and reduced cost (Kopp et al. 1997). A lab-on-a-chip for physiological fluids analysis is an example of microfluidic technology, once it can be a powerful tool in health care systems, helping professionals with rapid, accurate and sophisticated diagnostic tests for numerous substances. It can also be used by patients themselves for home diagnostic or monitoring tests. However, microfluidic technology also presents some challenges,

such as resistance to flow at micrometer dimensions and mixing difficulties in the laminar flow regime (Rife et al. 2000).

Therefore, several micromixing systems have been developed to overcome these limitations. Some solutions are based on MEMS (Micro Electro Mechanical Systems), such as micropumps and microvalves (Cambell and Grzybowski 2004). Despite its high efficiency, these solutions increase both the cost and the control electronics of the system and they are difficult to integrate into a single chip. Other approaches are the passive mixers which rely in mixing by diffusion. These result in long and complex channel topologies, which can be difficult to microfabricate and, usually, involve long transit and mixing times, in particular when the diffusion coefficients of the fluids are small (Ottino and Wiggins 2004). One mechanism that has received limited attention but seems to be able to overcome the aforementioned limitations is the use of acoustic streaming, which can be achieved by using a piezoelectric system. This phenomenon relies on a steady flow generated by the propagation of acoustic waves in fluids. It occurs due to the momentum and energy transfer from the acoustic field to the medium (Madelin et al. 2006).

In the last years, polymers have replaced traditional materials in several applications. A polymer that has received increased attention is poly(vinylidene fluoride) – PVDF. It shares its pyro- and piezoelectric characteristics, with an excellent combination of processability, mechanical and chemical resistance, and low production costs (Sencadas et al. 2006). These properties are at the origin of various applications, especially in the field of sensors and actuators (Lovinger 1982; Nalwa 1995). PVDF presents an unusual polymorphism among polymers, showing four



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different crystalline phases. From the technological point of view, the  $\beta$ -phase is the one which shows better properties for being applied in sensors, actuators and transducers, due to its higher piezo-, pyro- and ferroelectric properties (Davis et al. 1978; Martins et al. 2011).

Besides the excellent characteristics referred previously, electroactive PVDF has a set of properties that are very attractive when incorporated in microfluidic devices, such as transparency in the visible range and low acoustic and mechanical impedance, which is important and advantageous for the propagation of acoustic waves in fluids (Brown et al. 1992).

It must be noted that if the sound waves are propagated through fluids, most of the acoustic energy is reflected at the boundary layer between the piezoelectric material and the propagation medium. For that reason, piezoelectric materials with high-acoustic impedance will have a high reflection coefficient. This is the case of ceramic materials. However, despite the higher electromechanical coupling coefficients of the ceramic materials, their higher acoustic impedance results in a reflection coefficient on the boundary layer larger than 90%. Consequently, only a fraction of the acoustic energy generated by the piezoelectric element is transferred to the propagation medium (Foster et al. 2004). Moreover,  $\beta$ -PVDF is highly resistant to chemical agents and aging. While ceramic materials break easily and have hard and dense structures, PVDF is flexible, it has a low density and it can be easily produced into thin-films (Brown et al. 1992).

Other interesting feature of PVDF is its transparency, which increase the range of possible applications, like lab-on-a-chip that use optical detection (Fernandes et al. 2008). With this geometry, both the  $\beta$ -PVDF and the corresponding electrodes must be transparent to the visible light. Therefore, transparent conductive oxides such as ITO (Indium Tin Oxide) or AZO (Aluminum doped Zinc Oxide) must be used.

Summarizing, the incorporation of a piezoelectric film, such as PVDF in its  $\beta$ -phase, would be an advantage in a lab-on-a-chip device, once it can generate the proper acoustic streaming, improving the reaction of the fluids and, therefore, their mixing process. Further, it can also provide the necessary heating in specific reactions, as it will be seen later.

### ACOUSTIC STREAMING

The phenomenon of acoustic streaming was first analyzed in detail by Eckart in 1948 (Eckart, 1948) and overviewed by Nyborg in 1965 (Hamilton et al. 1998). Acoustic streaming can be used in several applications (Madelin et al. 2006), including chemical or pharmaceutical ones. Acoustic streaming is generated by the acoustic waves that can be produced when an electrical signal is applied to the contacts of a piezoelectric film. The absorption of the acoustic waves by the fluid results in a radiation pressure in the fluid itself along the direction of the acoustic propagation and attenuation (Figure 1) (Madelin et al. 2006; Rife et al. 2000). This absorption and the consequent motion causes the acoustic streaming, improving the mixture and the reaction of fluids.

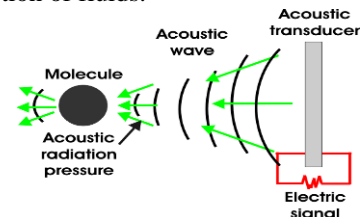


Figure 1: Generation of acoustics waves for fluids microagitation.

Further, when an electrical signal is applied to the piezoelectric film, just a small fraction of the electrical energy is converted into mechanical energy, whereas the remaining energy is converted into heat. For many applications including (bio)chemical reactions, the increase of temperature is advantageous, as it can reduce the reaction time. Thus, both the agitation and heating, i.e. acoustic thermoagitation, help to reduce the reaction time (Srinivasan et al. 2004).

### Preliminary studies

Taking into account that piezoelectric  $\beta$ -PVDF was never used for these type of applications, preliminary studies were performed to prove the feasibility of the acoustic streaming for accelerating the mixture of two fluids.

The quantitative evaluation of the mixing process was carried out using the *Far Diagnostic* kit and standards of urine with 5 mg/dl of uric acid concentration. The reagent reacts with the sample of urine containing uric acid in a 40:1 ratio with a maximum absorption at 550 nm.



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The acoustic streaming was studied for several frequencies and amplitudes of the electrical signal applied to the electrical contacts of a 110  $\mu\text{m}$  thick commercial  $\beta$ -PVDF film attached on one side of a regular cuvette. The system was calibrated and the reaction was studied by optical absorption spectrophotometry (SHIMADZU UV-3101PC) to a maximum time of 20 minutes, which is the time of the complete mixture without acoustic streaming.

First, acoustic streaming was performed actuating the  $\beta$ -PVDF, with a sinusoidal signal of 10 V amplitude at different frequencies. Then, a 10 MHz frequency sinusoidal signal with various amplitudes was applied. The results are shown in Figure 2.

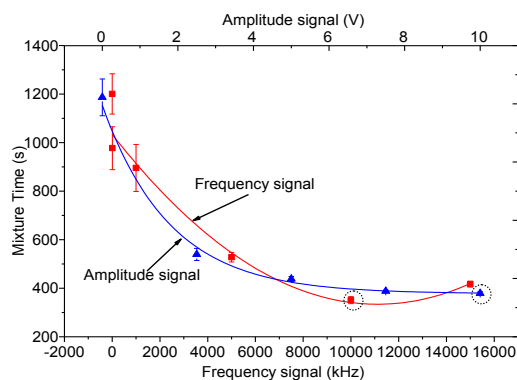


Figure 2: Mixing time as a function of the frequency and amplitude of the signal applied to the electrical contacts of a 110  $\mu\text{m}$  thick  $\beta$ -PVDF film.

It can be observed that with the application of acoustic streaming, the mixing occur in a faster way, being the time necessary to obtain the complete mixing, at 10 V amplitude and 10 MHz, only two sevenths (351 s) of the complete mixing time without acoustic streaming (1200 s). In this way, the resonance frequency of the used  $\beta$ -PVDF film (10 MHz) is the most efficient for the generation of acoustic streaming. This result is consistent with the theory (Eiras 2007). On the other hand, the mixing time decreases as the signal amplitude increases.

According to previous results, it was determined the individual contribution of the agitation and the heating generated by the use of the acoustic streaming technique. For that, the temperature profile of the sample was measured applying an electrical signal of 10 V amplitude and 10 MHz to the  $\beta$ -PVDF film. Then, the sample was heated with the same temperature

profile obtained before with the acoustic streaming, using a temperature controller (SHIMADZU TCC-260). It was observed that, for the two sevenths in terms of gains achieved by the application of the acoustic thermoagitation technique, three-fifths are due to heating and two fifths to agitation.

Proved the feasibility of  $\beta$ -PVDF based acoustic streaming at macroscale, the polymer films need to be designed and manufactured to allow their integration at microscale, which is not possible with the commercial films.

### PIEZOELECTRIC $\beta$ -PVDF POLYMER

As referred previously, PVDF can exist in four crystalline phases ( $\alpha$ ,  $\beta$ ,  $\gamma$  and  $\delta$ ). The  $\beta$ -phase is the one that exhibits the best ferroelectric and piezoelectric properties and a variety of methods like mechanical stretching (Sencadas et al. 2009), application of an electrical field (Davis et al. 1978) or incorporation of some additives (Martins et al. 2011) have been used to obtain this crystalline phase. However, spin-coating technique was chosen as it allows the fabrication of thin films over large areas with high structural uniformity on the desired substrates, avoiding any type of coupling that can limit the passage of sound waves generated to the fluids and consequently a decrease of the efficiency of the acoustic streaming phenomenon (Norrman et al. 2005). The  $\beta$ -phase is obtained due to the stretching of the polymer chains generated by the shear and elongation forces during spin-coating deposition.

The development of sensors and actuators requires obtaining high quality films with flat and smooth surfaces and uniformity in terms of thickness and electroactive phase. A study of how the parameters involved in the processing of PVDF by spin-coating influence the properties of the films was carried out. In this way, sensors and actuators based on PVDF could be manufactured with controlled thickness, morphology and electroactive phase content, so that the material can be designed for specific applications, including acoustic streaming for lab-on-a-chip.

### Processing

PVDF in powder form (Solef 1010, Solvay) was dissolved in N,N-Dimethylformamide (DMF) (Aldrich, analytical grade) using a magnetic stirrer. Heating at 30°C was used in the first 15 min of the mixture to help dissolving and prevent agglomerates. PVDF films were



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deposited by spin-coating (Laurell WS-6505-6NPP/A1/AR2) on highly polished glass substrates. Room temperature and humidity were 20°C and ~ 50%, respectively. The morphology of the films was studied using a scanning electron microscope (SEM) NanoSEM-FEI Nova 200 and their transmittance using an optical spectrophotometer (Shimatzu UV-3101PC). Films thickness was measured using a profilometer (Veeco Dektak 150) and a digital micrometer (Fischer Dualscope 603-478). The crystalline phases of the films were determined by infrared spectroscopy (FTIR Spectrum 100) with ATR system (FTIR-ATR) and confirmed by X-ray diffraction (XRD, Bruker D8 Discover). Piezoelectric response was measured with a  $d_{33}$ -meter APC YE2730A.

### Results and Discussion

The influence of thermal annealing on the morphology and surface quality of the PVDF films was studied. As referred previously, the aim is to obtain films with a smooth and flat surface. A solution with 20%wt PVDF was deposited by spin-coating (angular velocity, acceleration and time of 1000 rpm, 750 rpm/s, 30s, respectively) and then dried at different temperatures between 20°C (room temperature) and 80°C using a hot-plate (Präzitherm PZ23-2). It is noticeable the difference in porosity and optical transparency between the samples dried at room temperature and those dried at higher temperatures (Figure 3). At room temperature, the evaporation rate of DMF is slower, yielding to the formation of pores (Magalhães et al. 2011) and films with transmittance below 20% across the visible range. This porous structure hampers the deposition of conducting electrodes, hinders the poling process and the electroactive response of the material, and prevents applications in which transparency of the material is required. A thermal annealing between 30°C and 80°C increases the rate of solvent evaporation avoiding the formation of pores, leading to films with a smooth and flat surface and with a transmittance higher than 70% across the visible range. It is also to notice the thickness difference between porous (Figure 3a) and nonporous films (Figure 3b): 9µm and 4.5µm, respectively. Annealing the samples between 30°C and 80°C does not lead to any further variation in films thickness. On the other hand, by annealing at 80°C or higher temperatures, the nonelectroactive  $\alpha$ -phase of the polymer is obtained, as revealed by the formation of a

ringed-spherulitic microstructure and confirmed by FTIR (Figure 4a).

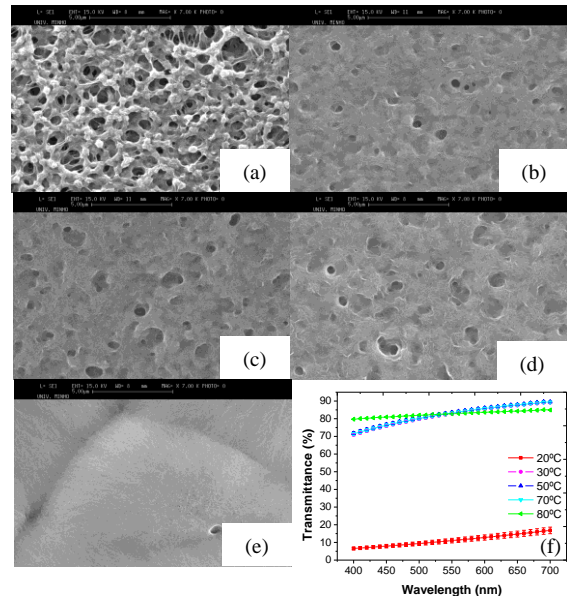


Figure 3: SEM micrographs of the PVDF films (a) without post-thermal annealing; with post-thermal annealing at: (b) 30°C, (c) 50°C, (d) 70°C, (e) 80°C; (f) Transmittance of the above films.

Figure 4 shows the FTIR-ATR spectra of the films annealed at temperatures from 20°C to 80°C. Taken into consideration specific absorption bands, 763 and 839  $\text{cm}^{-1}$ , ascribed to the  $\alpha$ - and  $\beta$ -phases, respectively, the  $\beta$ -phase fraction present in the films is calculated (Salimi et al. 2003). Figure 4b shows the  $\beta$ -phase content of the samples and their piezoelectric coefficients after poling by corona. It is observed a decrease of the  $\beta$ -phase content with increasing annealing temperature due to the increase of the solvent evaporation rate and faster crystallization of the film, which leads to preferential formation of  $\alpha$ -phase (Chinagli et al. 2010). In this way, smooth and flat PVDF films ~4 µm thick and with  $\beta$ -phase content from 0 to 75% can be obtained just by varying the thermal annealing temperature. It is to notice that 75 to 85% is the maximum amount of  $\beta$ -phase in commercial films. Moreover, the higher the  $\beta$ -phase content, the higher the piezoelectric coefficient of the film (Gomes 2010) (Figure 4b). The electrical poling of the PVDF films was performed by a corona discharge. The applied





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voltage was 10 kV at a constant current of 15  $\mu$ A. The distance between the sample and the tip was 2 cm and the poling temperature 100°C. The films were subjected to the corona field for 45 minutes and were cooled down to room temperature with the electrical field applied. For a thermal annealing at 30°C, a maximum value of the piezoelectric coefficient of -21 pC/N with a  $\beta$ -phase content of 75% was obtained (Figure 4b). With increasing annealing temperature, a continuous decrease of the  $\beta$ -phase content and the piezoelectric response is observed until the thermal annealing at 80°C in which the  $\alpha$ -phase content and the piezoelectric response are zero.

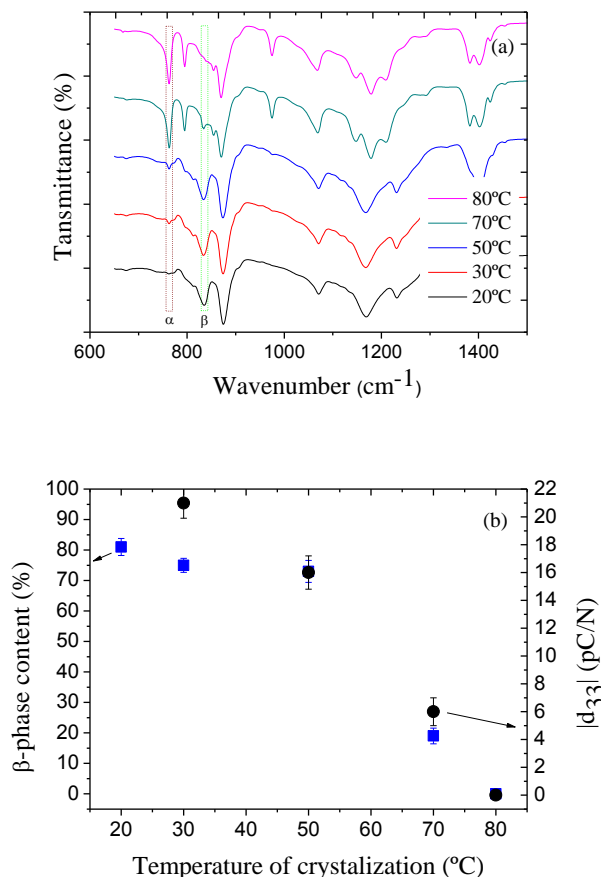


Figure 4: (a) FTIR-ATR spectra of films subjected to various post-thermal annealing; (b)  $\beta$ -phase content and the correspondent piezoelectric coefficients module of the PVDF films as a function of the thermal annealing.

In addition to the piezoelectric response, it is also important to control the thickness of the films. This was achieved by preparing samples from PVDF/DMF solutions with 10, 15 and 20 %wt of PVDF at spin-coater speeds ranging from 1000 to 8000 rpm. All samples were further subjected to a thermal annealing at 30°C to obtain the best electroactive characteristics and the maximum transmittance. Figure 5a shows that the higher the angular velocity of the spin-coater and the lower the viscosity of the solution, the thinner the samples. In this way, PVDF films with a thickness from 300 nm to 4.5  $\mu$ m can be obtained by adjusting the PVDF/DMF mass ratio and the angular velocity of the spin-coater. Thinner films cannot be obtained by this method as there is a compromise between the concentration of DMF and the evaporation rate of the solvent which in turn depends on the angular velocity during spin-coating deposition. For larger DMF contents, the viscosity is lower and the film crystallizes in a porous structure. Thinner films can be eventually obtained by the incorporation of a heating system within spin-coater [9]. Thicknesses larger than 4.5  $\mu$ m are also difficult to obtain by increasing the viscosity of the solution. For higher polymer mass ratios, the dissolution of PVDF involves the use of heating, which can affect the properties of the obtained films [17]. Finally, the effect of the rotational velocity on the  $\beta$ -phase content and piezoelectric coefficient of the PVDF films was studied. As referred previously, a higher evaporation rate favored formation of the  $\alpha$ -phase. So, it could be expected an increase in the  $\alpha$ -phase content with increasing rotational velocity and decreasing PVDF/DMF mass ratio. However, as shown in Figure 5b, the higher the rotational speed and the lower the PVDF/DMF mass ratio, the higher the  $\beta$ -phase content of the films, with  $\beta$ -phase contents up to 100%. This can be explained by the stretching of the polymer chains generated by the shear and elongation forces during spin-coating deposition, which superimposes the effects of solvent evaporation rate and it promotes the  $\alpha$  to  $\beta$ -phase transformation (Sencadas 2009). The higher the rotational speed, the larger is the stretching effect and therefore the  $\beta$ -phase content. No significant variation of the piezoelectric response is observed with the variation of the angular velocity of the spin-coater.



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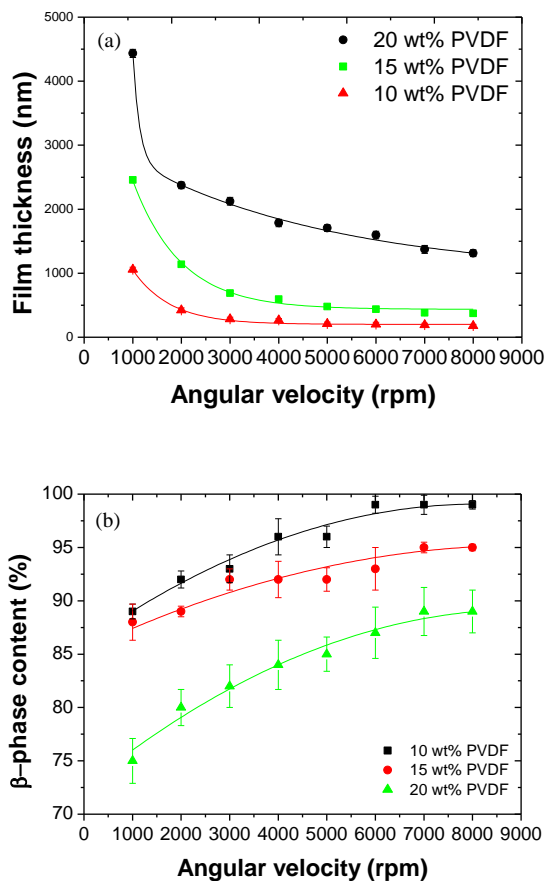


Figure 5: Film thickness (a) as a function of the angular velocity of the spin-coater for various mass fractions of PVDF/DMF and a thermal annealing of 30°C and (b) the corresponding  $\beta$ -phase content.

### CONCLUSIONS

It was proven that the application of acoustic streaming based on a piezoelectric commercial  $\beta$ -PVDF improves mixing and reaction time, making this technique suitable for improving the performance of fluidic devices. However, the aim objective is to use this technique at the microscale to enhance microfluidic mixing efficiency in lab-on-a-chip devices. Consequently, the manufacturing process of PVDF was studied and optimized for use at this scale.

A simple and reproducible way to prepare electroactive PVDF samples for sensor and actuator applications with

controlled thickness from 300 nm to 4.5  $\mu\text{m}$ , electroactive phase content from 0 to 100%,  $d_{33}$  piezoelectric coefficients from 0 to -21 pC/N and optical transmittance up to 70% in the visible range was presented. The control of the PVDF films has been achieved by varying the thermal annealing temperature, the angular velocity of the spin-coating process and the PVDF/DMF mass ratio.

Experiments have been performed at the microscale with promising results. Further directions are to control the acoustic streaming and the heating generated, according to the dimensions of the channels, dimensions of the piezoelectric  $\beta$ -PVDF polymer and the actuation electrical signal. These features allow applying this technique in large range of lab-on-a-chip devices with one purpose: increase their mixing efficiency.

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