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Using Layer-by-Layer Self-Assembly in the fabrication of thin films

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Abstract: **The latest innovation in the field of nanoscience demonstrate integration of nano architectonics with the Layer-by-Layer (LBL) technique. The LBL technique is leveraged in this study to perform thin film fabrication using poly (diallyl dimethylammonium chloride) (PDDA) and copper phthalocyanine-tetrasulfonic acid (CuTsPc). The deposition of PPDA/CuTsPc films over a glass substrate through alternate adsorption is studied in this research. Both manual and automated methods are examined to evaluate the efficiency of the LBL technique. Ultraviolet-visible spectroscopy and Atomic Force Microscopy are used to monitor the film deposition. The results indicate that this method is viable in terms of affordability and reproducibility even without exercising control over parameters such as pH and temperature.**

Keywords: **Layer-by-Layer self-assembly, poly (diallyl dimethylammonium chloride) (PDDA), copper phthalocyanine-tetrasulfonic acid (CuTsPc), Atomic Force Microscopy, UV-Vis spectroscopy, Nanoscience, Nanotechnology.**

1. INTRODUCTION

Nanoscale is a magical point on the dimensional scale; structures in nanoscale (called nanostructures) are considered at the borderline of the smallest of human-made devices and the largest molecules of living systems[1]. According to literature, the nanoworld has emerged from the combination of science and technology. A substantial number of new materials with nano elements such as ceramics, glass, polymers and fibers are making their way onto the market and are present in all shapes and forms in everyday life, from washing machines to architecture[2].

The latest areas of innovation in nanoscience and nanotechnology savor integration the Layer-by-Layer (LBL) technique with nano architectonics. Recent advances in LbL assembly technologies have explored different driving forces for the assembly process when compared with the diffusion-driven kinetics of classical LbL assembly, where a substrate is immersed in a polymer solution[3].

The LBL is a novel method for preparing charged thin films by alternate adsorption. These films are fabricated by forming alternate layers of oppositely charged materials that are infused with washing procedures. The principle of this technique is the charge reversal of the film layer during each dip which makes the surface alternate charge reversal. The film is deposited on glass/gold or quartz substrate by dipping the slide into a beaker containing polycation, manually or by automated device, and there by making the substrate positively charged (i.e. the slide positive) and then multilayered films are formed by the adsorption of polyanion and polycation successively [4].

This research paper serves as an analytical lens for the LBL technique, involving the deposition of three films of PDDA/CuTsPc over a glass substrate. A comparison is drawn between manual and automated fabrication to study the technique without exercising control over parameters such as pH, conductivity, turbidity, and temperature.

2. METHODOLOGY

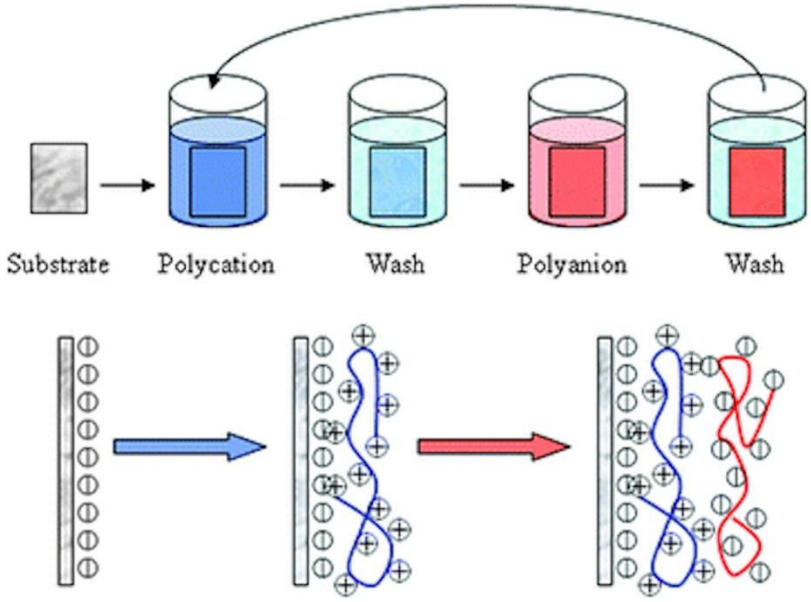

Fig. 1: Scheme of sample preparation by LBL

A clean glass substrate is used to perform the Layer by Layer assembly technique for fabricating three thin films of PDDA/CuTsPc. The first sample is prepared manually by immersing the glass substrate in an aqueous solution of polycation for eight minutes, which is followed by washing in deionized water and drying using nitrogen gas. This process is repeated until five bilayers are formed. UV-Vis spectroscopic measurements are used to analyze the absorbance trends. For the second sample, the same process is repeated but with an automated apparatus. An aqueous polyanion solution and an automated apparatus is used for the third sample with the remaining steps being identical to that of the previous samples.

The poly(diallyldimethylammonium chloride) 2 (PDDA) was used as the polycation and copper phthalocyanine-tetrasulfonic acid (CuTsPc) as the polyanion for depositing the thin films.

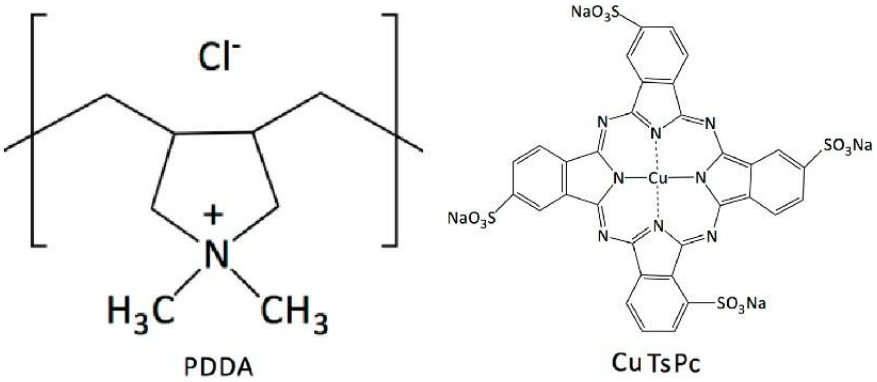

Fig. 2: Structural formulas of PDDA and CuTsPc

Due to the strongly charged structures of PPDA bearing a low molar mass, it causes high repulsion between the layers for the formation of LBL films, thereby producing films as thin as 1.7 nm. A metal ion can replace the central hydrogen atoms of the phthalocyanines which usually comprises two hydrogen atoms. Phthalocyanines are insoluble in water, so, sulphonic groups SO are added to the benzene rings to that facilitate the reaction in water. It results in the production of copper phthalocyanine-tetrasulfonic acid. Thin films of the order 0.6 nm are produced by the phthalocyanine. Atomic Force Microscopy (AFM) has been used to examine the thin films. UV-Vis spectroscopy is employed to investigate the bilayers at every deposition.

3. RESULTS

UV-Vis Spectroscopy Results

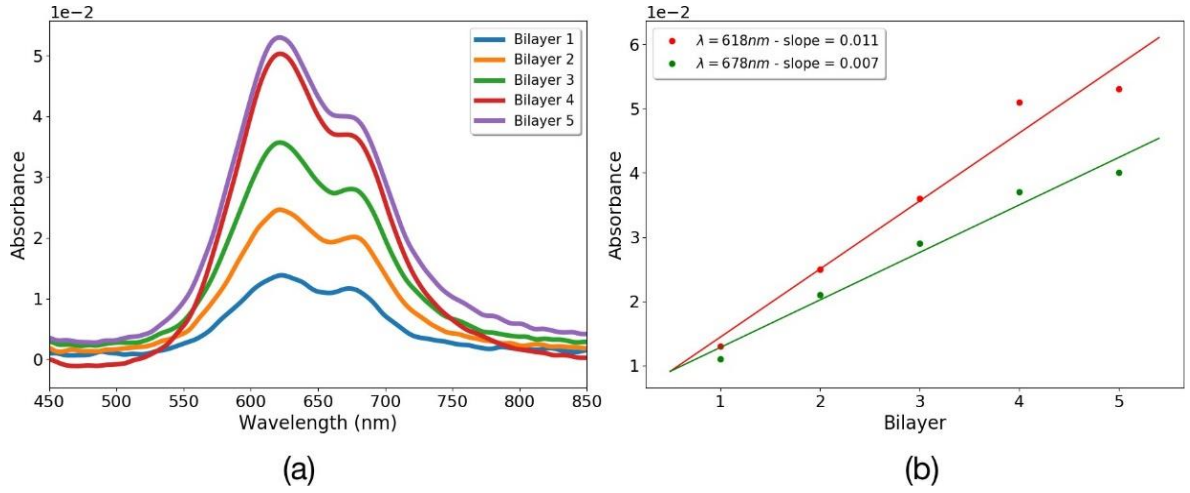

Fig. 3: (a) UV-Vis absorption spectra of glass with PDDA/CuTsPc film for various bilayers. (b) Linear graph of the absorbance at 618 and 678 nm observed as a function of bilayers of PDDA/CuTsPc.

UV-Vis spectroscopy is applied to analyze the absorption trends. Figure 3(a) demonstrates UV-Vis absorption spectra of glass covered with about one to five bilayers of PDDA/CuTsPc for the manually prepared samples. It is seen that the increase in peaks are directly proportional to increase in the number of bilayers. This shows that adsorption of PDDA or CuTsPc occurs at every step of film deposition. The linear behavior of the absorption spectra in Figure 3(b) indicates the orderly nature of the film fabrication process

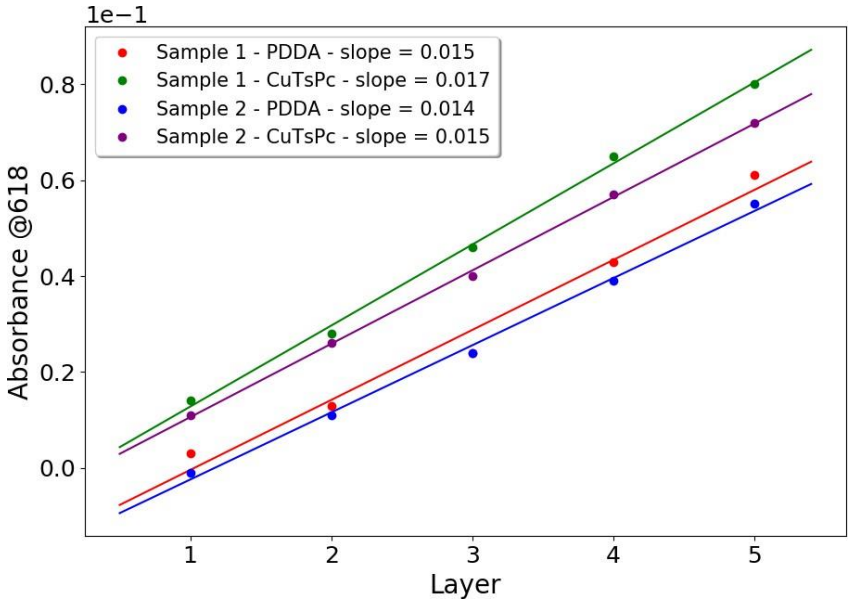

Fig. 4: UV-visible absorption spectra of glass substrate coated with PDDA/CuTsPc thin film for different number of bilayers.

Figure 4 demonstrates the linear behavior for the samples prepared by automated fabrication. The linear growth of the samples is fairly alike for the monolayers of PDDA and CuTsPc. Such a similarity validates the reproducibility of the films through the methods used in this study without having to exercise control over any of the parameters.

Atomic Force Microscopy Results

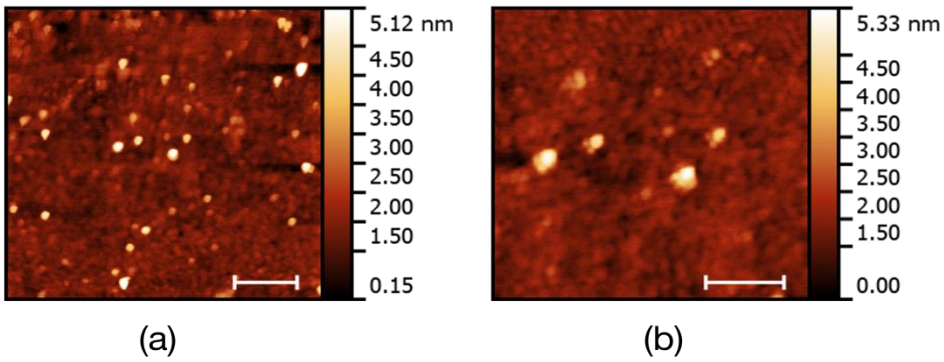

Fig. 5: AFM images of the surface of the glass substrate before film fabrication. Scale bar: (a) 1µm and (b) 500nm.

Figure 5 demonstrates AFM images of the glass prior to film fabrication for various scan sizes. To ensure that the materials are being adsorbed, the images of the substrate before and after film deposition are compared.

The roughness Root Mean Square (RMS) values calculated from Figure 5(a) is are (479 ± 24)pm and from Figure 5(b) is (413 ± 20)pm

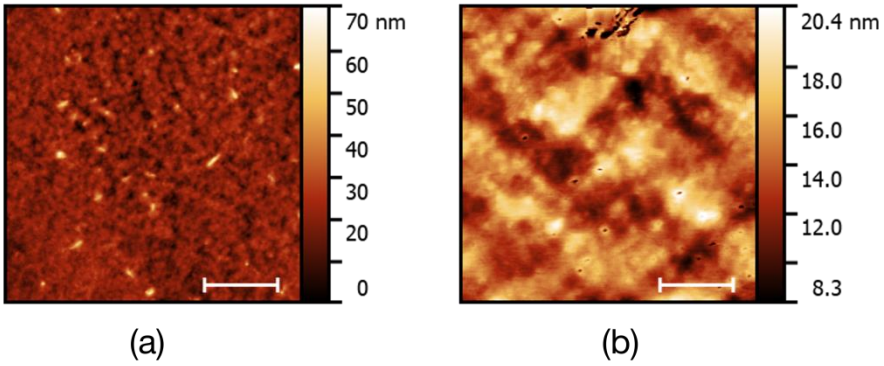

Fig. 6: AFM images of the surface of the glass substrate after PDDA/CuTsPc film deposition. Scale bar: (a) 1µm and (b) 500nm.

Figure 6 shows AFM images of the substrate after it is coated with 15 bilayers of PDDA/CuTsPc. The difference in the surface images can be easily observed by comparing Figure 6 with Figure 5. Figure 6 contains images of the surface topography of PDDA/CuTsPc films with an area 20 x 20 µm for Figure 6(a) and 2 x 2 µm for Figure 6(b).

The roughness RMS values obtained from Figure 6(a) is (5.0 ± 0.3)nm and form Figure6(b) is (3.4 ± 0.2)nm.

Upon analyzing the differences between figure 5(b) and 6(b), it is observed that the LBL films are formed on the substrate as a consequence of the change in topography. RMS values also savor changes in roughness values which increase substantially after film deposition that is (413 ± 20)pm to (3.4 ± 0.2)nm.

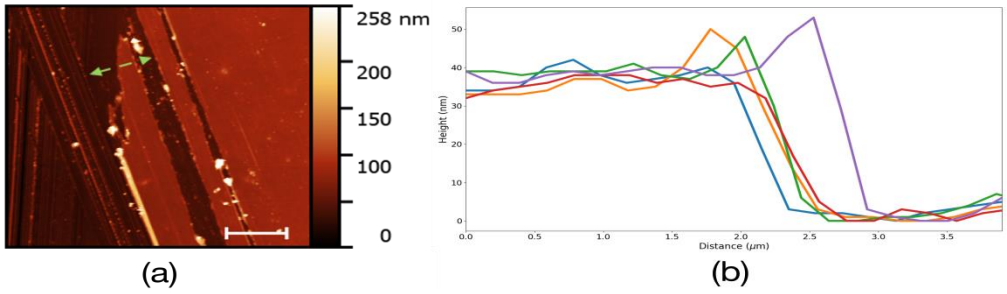

Fig. 7: (a) AFM images showing a stage of PDDA/CuTsPc film deposition. Scale bar: 10 µm (b) Height profile of this step

In Figure 7(a), an AFM image of one of the film fabrication steps is. This topography image helped in determining the film thickness which can be seen in Figure 7(b). The curves shown in figure 7(b) have an average thickness $\bar{T}$ = (33±2)nm. The thickness of PDDA monolayer is estimated to be. 1. 72 nm[5] and the thickness of CuTsPc monolayer is ~0.6 nm[6]. This data is not analogous to the thickness measured for the 15 PDDA/CuTsPc bilayer thickness, (33±2)nm. It demonstrates that this method is genuinely depositing monolayers at every stage of the film fabrication process.

4. CONCLUSION

The Layer-by-Layer (LBL) technique is widely regarded as one of the best thin film fabrication techniques. It enables the fabrication of ultra-thin films that can embed materials on the nanoscale, which finds substantial applications in nanoscience and nanotechnology. In this research, two methods for thin film fabrication are presented using the LBL technique. Glass substrate is used for the experiments which are coated with PDDA/CuTsPc films to study absorbance. An orderly deposition process is observed upon performing Atomic Force Microscopy and UV-Vis spectroscopy. The research findings show a linear behavior in the absorbance curve, which demonstrates the fact that monolayers form at every stage of the film fabrication process. It is also observed that the materials were being absorbed at the same rate for both the automated and manual methods savoring that LBL is both an affordable and reproducible technique for thin film fabrication

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