THE PATENTS ACT 1970

COMPLETE SPECIFICATION

SECTION 10

A METHOD OF MANUFACTURE OF A DEVICE TO MONITOR THE FLOW OF LIQUIDS AND A DEVICE MANUFACTURED BY THE SAID METHOD

INDIAN INSTITUTE OF TECHNOLOGY, IIT P.O., CHENNAI 600 036, TAMIL NADU, INDIA, AND INDIAN AUTONOMOUS BODY SET UP BY THE GOVERNMENT OF INDIA UNDER AN ACT OF PARLIAMENT

THE FOLLOWING SPECIFICATIONS PARTICULARLY DESCRIBES THE NATURE OF THIS INVENTION AND THE MANNER IN WHICH IT IS TO BE PERFORMED

This invention relates to a method of monitoring the flow of liquids and a device manufactured according to the said method.

The method herein proposes to fabricate a gold nanoparticle multilayer assembly on an Indium doped tin oxide conducting glass substrate and a device to monitor the flow of liquids. This technology is believed to have far-reaching applications in nanofluidics and in monitoring the flow of liquids in biological channels. It is possible to construct such flow devices on various kinds of substrates with other nanoparticles for measuring flow of pure and impure liquids.

Scientists' and technologists' worldwide focus on the possible use of nanoparticles as sensors due to the fact that they offer remarkable sensitivity and selectivity for the analyte species. Their ability to detect very low amount of the analyte arises from their large surface to volume ratio.

The nanoparticles of gold, stabilized by citrate groups (Au@citrate) were synthesized by a procedure reported in the literature. The typical size of the particles synthesized by this method is 15-20 nm in diameter.

The method according to this invention comprises the steps of synthesizing the nanoparticles of gold, stabilized by citrate groups; cleaning a substrate and sonicating with deionised water; activating the surface of the substrate by soaking in 10% solution of HCl; washing the substrate followed by drying the same in an oven at 100^0 C for 1 hour; annealing the same thereafter in a hot air furnace at 450^0C for 7 hours; cooling and drying the substrate in a desiccator; coating the substrates with a multilayer assembly of gold nanoparticles by dipping the substrate in a 3 % solution of 3-aminopropyl triethoxy silane (anchor molecule) in 2-propanol, for 2- 5 minutes; washing the substrate with 2-propanol; heating the substrate at 110^0C for 10 minutes to remove 2-propanol; cooling and drying the substrate in a desiccator; dipping the substrate in an aqueous solution of Au@citrate for 10-15 minutes; removing the substrate, washing with water and dipping the substrate in 2-propanolic solution of 1,6-hexanedithiol (spacer molecule) for 30 minutes. Dipping in Au@citrate and 1,6 hexanedithiol are repeated to achieve the desired number of layers; and finally dipping the substrate in a spacer molecule to obtain the device.

The invention proposed herein will be described with reference to the accompanying drawings wherein,

Fig.1 shows the schematic of the experimental set-up illustrating the direction of the flow, Fig. 2 shows the potential versus time for the sensor device, and Fig. 3 shows plots of potential difference as a function of (A) flow rate (B) dipole moment and (C) ionic concentration.

A commercially available one-side indium doped tin oxide conducting glass (ITO) plate was used as the substrate. Besides being transparent, it is possible to measure electrical properties across this plate. The substrate was first cleaned with mild detergent solution to remove greasy impurities. It was then repeatedly cleaned and sonicated with deionised water. Soaking in 10% solution of HCl activates the surface of the plate thus cleaned. The plates are then washed with water and dried in an oven at 110^{0}C for 1 hour, annealed in a hot air furnace at 450^{0}C for 7 hours, cooled in a desiccator and preserved in the same until further use.

A multilayer assembly of gold nanoparticles was made as follows. The ITO plate was dipped in a 3% solution of 3-aminopropyl triethoxy silane (anchor molecule) in 2-propanol, for 2-5 minutes. The anchor molecules act as a link between the substrate and the nanoparticle assembly and facilitate the adhesion of the multilayer assembly to the substrate. The next step involves the heating of the plates at 110^{0}C for 10 minutes to remove 2-propanol. This is followed by cooling inside a desiccator, and then dipping in aqueous solution of Au@citrate for 10-15 minutes. After this, the plate was removed, washed with water and subsequently with 2-propanol to remove unabsorbed species. The plate was then dipped in a 30mM solution of 1,6-hexanedithiol (spacer molecule) in 2-propanol. The spacer molecules help in separating the successive nanoparticle layers and thus a clear demarcation can be made between thin films and the multilayer assembly fabricated here. Dipping in Au@citrate and 1,6-hexanedithiol is repeated to achieve the desired number of layers (three, in our experiments). A final dipping in the spacer molecule is done to ensure the stability of the nanoassembly by developing cross links between the gold nanoparticles.

In the set-up used for the studies, we pass the desired liquid through a cylindrical glass tube of 22 mm inner diameter and 36 cm length within which the sensor device was suspended as shown in Fig.1. A peristaltic pump regulates the flow. The liquid flows against the gravity, and care was

taken so that no air bubble was trapped within. The electrical connections from the plates are taken out of the flow device through insulated copper wires and the potential was measured by a Keithley 2700 multimeter/data acquisition system. During the experiment, the plate containing the multilayer assembly was connected to the negative of the voltmeter and a positive reading was obtained. This confirms the accumulation of electrons on the plate containing the multilayer assembly. In a typical measurement, after stabilization of the whole set-up, which takes 500 – 1000 s, the plates were shorted and the reading was collected subsequently for a period of 4000 s. The device was shorted again for a period of 100 s and the measurements were repeated. Water used was triply distilled in a glass apparatus. Solvents were distilled prior to use. Fig.1 shows a schematic of the experimental setup showing the direction of flow. The cylindrical column housing the sensor device has been shown in part. The sensor device has been blown up on the right to show a schematic of the multilayer assembly indicating the nanoparticles, the anchor and the spacer molecules.

Specific example of the working of the device

Experiment with water

A typical voltage response over many such cycles is shown in the inset of Fig. 2. Curve (a) in Fig. 2 shows the initial response of the device when water is passed at a flow rate of 500 cc/h over one cycle of the measurement. The potential difference saturates to 18.3 mV within 1000 s. The response can be contrasted to that of curve (b) in the same figure that shows the voltage generated with the uncoated plates, which confirms that the voltage response is only due to the nanoparticle layer.

Other experiments

In order to understand the role of several other parameters influencing the generation of the potential difference, experiments were done with liquids of varying dipole moment, viscosity and ionic concentration. All these measurement were conducted for a flow rate of 500 cc/h for the liquids, 2-propanol, water, acetone and acetonitrile with dipole moments of 1.56, 1.85, 2.88 and 3.92 D, respectively. The dependence of potential difference on the viscosity of the flowing analyte is arrived at by testing the liquids of varying ratios of water:2-propanol mixtures. For studying the

effect of ionic concentrations, aqueous solutions of trisodium citrate was used. The relevant data for all these experiments are given in Fig. 3.

Control experiment

To further confirm the effect of flow, the experiment was done with the sensor device being suspended in still water. The response observed in this case was of the order of 1-3 mV, reiterating the fact that the moving liquid is responsible for the potential. We theorise that the potential is generated by knocking out of charges from the nanoparticle surface by the coulomb collision of flowing dipoles.

We claim:

1. A method of manufacture of a device to monitor the flow rate of liquids comprising the steps of synthesizing the nanoparticles of gold, stabilized by citrate groups; pre-cleaning of the indium doped tin oxide conducting glass (ITO) by sonicating with deionised water; activating the surface of the substrate by soaking in 10% solution of HCL; washing the substrates with water followed by drying the same in oven at 100^0C for 1 hour; annealing the same thereafter in a hot-air furnace at 450^0C for 7 hours; cooling and drying the substrate in a desiccator; coating the substrates with a multilayer assembly of gold nanoparticles by dipping the substrate in a 3% solution of 3 – aminopropyl triethoxy silane (anchor molecule) in 2 – propanol for 2-5 minutes; heating the substrate at 110^0C for 10 minutes to remove 2-propanol; cooling and drying the substrate in a desiccator; dipping the substrate in an aqueous solution of **Au@citrate** for 10-15 minutes; removing the substrate, washing with water; dipping in **Au@citrate** for 10-15 minutes; washing with water followed by 2-propanol; dipping in a 2-propanolic solution of 1,6 hexanedithiol (spacer molecule) for 30 minutes. dipping in **Au@citrate** and 1,6 hexanedithiol are repeated to achieve the desired number of layers; and finally dipping the substrate in the solution of the spacer molecule.

2. A method as claimed in claim 1 wherein the substrate is a one-side conducting indium doped tin oxide conducting glass plate or any other suitable substrate with which an electrical potential can be measured.

3. A method of manufacture of a device to monitor the flow rate of liquids as herein deecribed and illustrated.

Dated this the 11th day of August 2005.

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ABSTRACT

1113/CHE/2005

Title : A Method of Manufacture of a Device to Monitor the Flow Rate of Liquids

A method of manufacture of a device to monitor the flow rate of liquids comprising the steps of synthesizing the nanoparticles of gold, stabilized by citrate groups; pre-cleaning of the indium doped tin oxide conducting glass (ITO) by sonicating with deionised water; activating the surface of the substrate by soaking in 10% solution of HCL; washing the substrates with water followed by drying the same in oven at 100 C for 1 hour; annealing the same thereafter in a hot-air furnace at 450 C for 7 hours; cooling and drying the substrate in a desiccator; coating the substrates with a multilayer assembly of gold nanoparticles by dipping the substrate in a 3% solution of 3 – aminopropyl triethoxy silane (anchor molecule) in 2 – propanol for 2-5 minutes; heating the substrate at 110 C for 10 minutes to remove 2- propanol; cooling and drying the substrate in a desiccator; dipping the substrate in an aqueous solution of **Au@citrate** for 10-15 minutes; removing the substrate, washing with water; dippingin **Au@citrate** for 10-15 minutes; washing with water followed by 2-propanol; dipping in a 2-propanolic solution of 1,6 hexanedithiol (spacer molecule) for 30 minutes. Dipping in **Au@citrate** and 1,6 hexanedithiol are repeated to achieve the desired number of layers; and finally dipping the substrate in the solution of the spacer molecule. (Figure 1)

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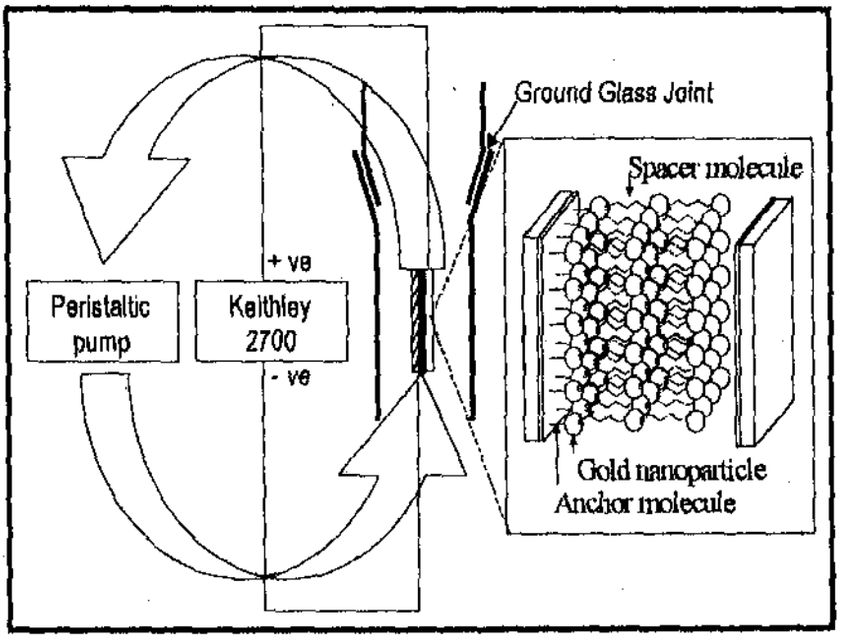

Fig. 1

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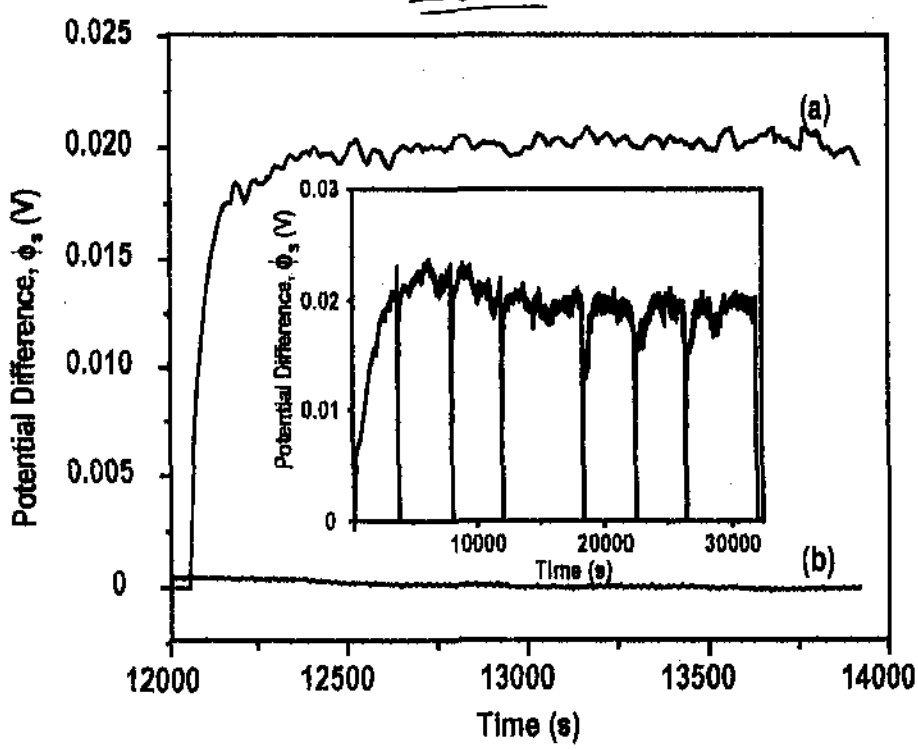
Fig. 2



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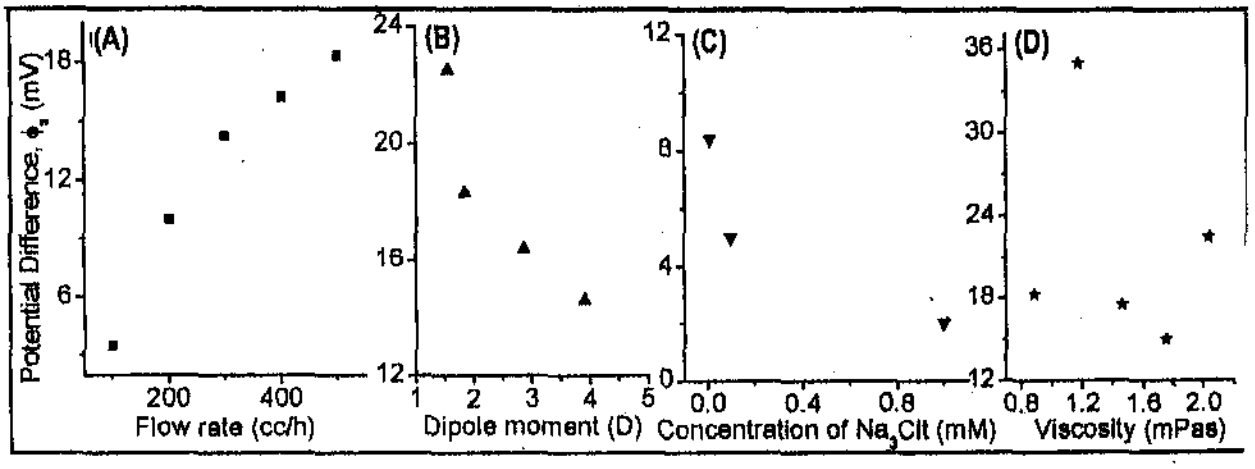

Fig 3

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