Nanobio Technology Enabled Point-of-Care Devices
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Lecture - 39
Lab Demonstration - 2

Hello students. Today we are going to briefly demonstrate nanoparticle synthesis using different methods of different nanoparticles. So, today basically we will show you two different nanoparticle synthesis methods. One, weight chemical synthesis of gold nanoparticles and secondly, we will show you the electrochemical synthesis of the copper nanoparticles.

So, nanoparticles are basically very small in nanodiameter range that is 1 to 100 nanometer in size that is one tenth billion of a meter. So, why nanoparticles are important in our case for electrochemistry or electrochemical sensor? Because the nanoparticles increases the surface area which increases the catalytic activity of the electrode, which basically increases the signal to background ratio of our particular signal.

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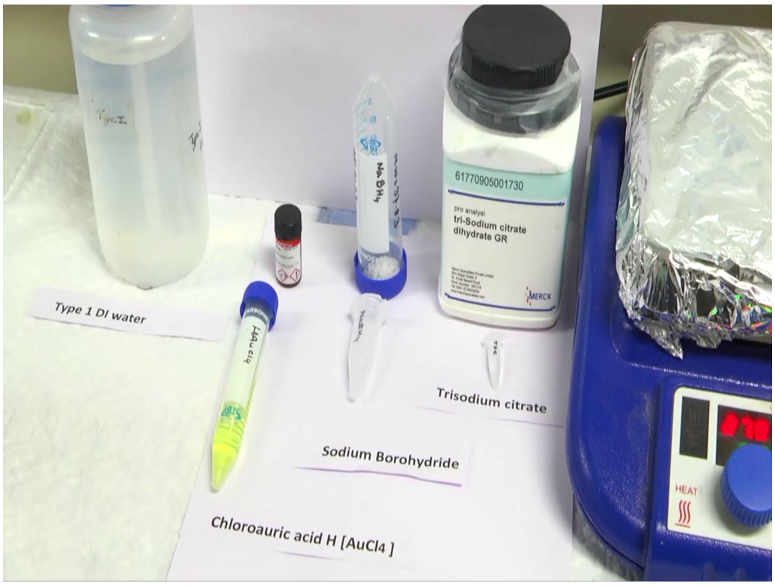

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Now, I will show you the weight chemical synthesis of gold nanoparticles. So, for that we will need gold nanoparticle precursor as chloroauric acid and then we will need sodium borohydride and also the trisodium citrate. Here the sodium borohydride will act as an reducing agent and trisodium citrate will act as a capping agent which creates a electrostatic sheets around the nanoparticle which reduces the agglomeration of the nanoparticles.

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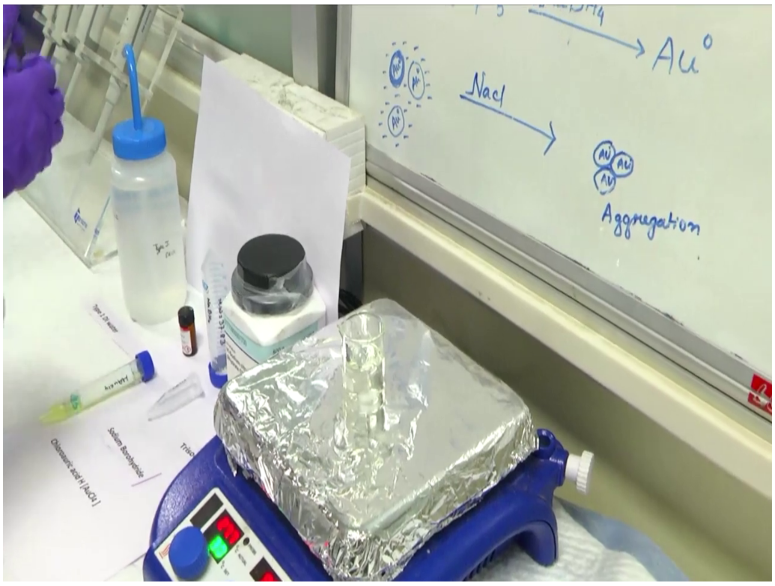

So, for the synthesis we will need 20 ml of type 1 di water which will be placed over a magnetic stirrer as we need continuous stirring of during the synthesis and in magnetic stirrer, we will set 900 rpm. Now, I will add 1 millimolar of 1 ml HAuCl4 solution in this stirring type 1 di water. After that we will add 200 micro litre of capping agent in that solution.

We will wait for some time. After some time, we will add drop by drop sodium borohydride solution.

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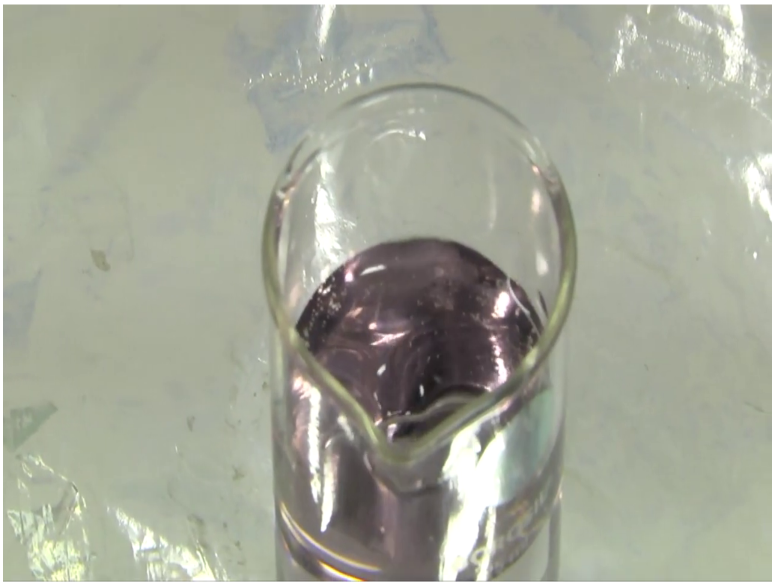

After adding we can see the colour changing.

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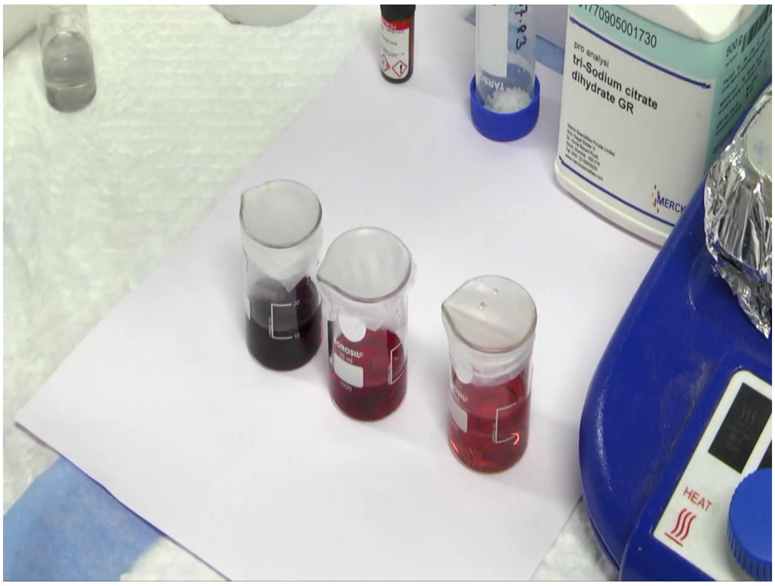

From this colour change we can tell that the nanoparticles is formed. So, now I will briefly tell you the theory behind this colour change due to the nanoparticle formation. So, when the gold nanoparticles in different size in solution phase interacts with the visible light and creates different colour.

So, the oscillating electric field of the visible light when reacts with the gold nanoparticles electron charge it creates a oscillation which resonates with the visible light energy and this phenomenon is known as surface plasmon resonance. So, when the nanoparticles are very small in size this colour at this surface plasmon phenomenon creates a absorption of the light is blue green region and reflects the red light.

When the nanoparticle size gradually increases here, we can see this is light red colour and this converted to ruby red colour and then it is wine red colour. So, when the nanoparticle size increases it absorbs the light in a red region and reflects a blue region. So, we can see pale blue to purple colour also wine red colour we can see.

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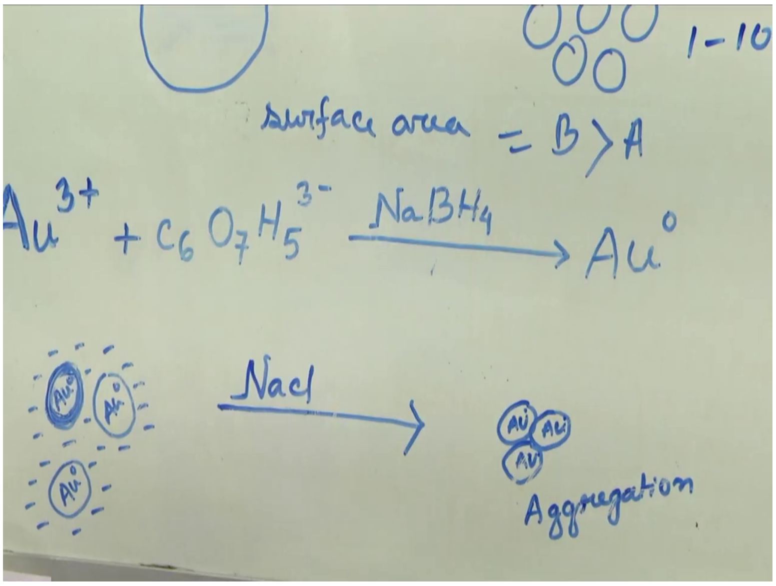

Now, we will see the addition of salt effect on that formed nanoparticles. So, this is our red coloured nanoparticle that is formed. So, when we will add NaCl it disrupt the electrostatic sheet around the nanoparticles and create aggregation. So, in this I have added the NaCl and we can see here the aggregation of the gold nanoparticles and here you can clearly see some large particle size.

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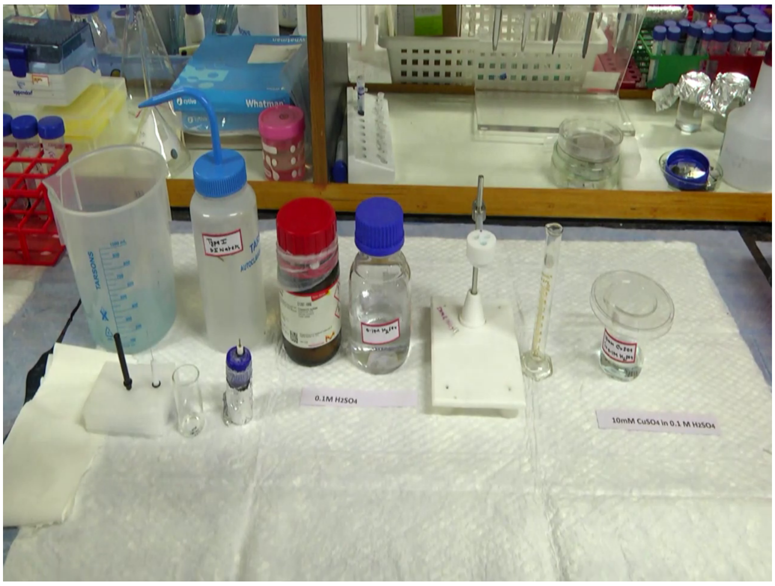

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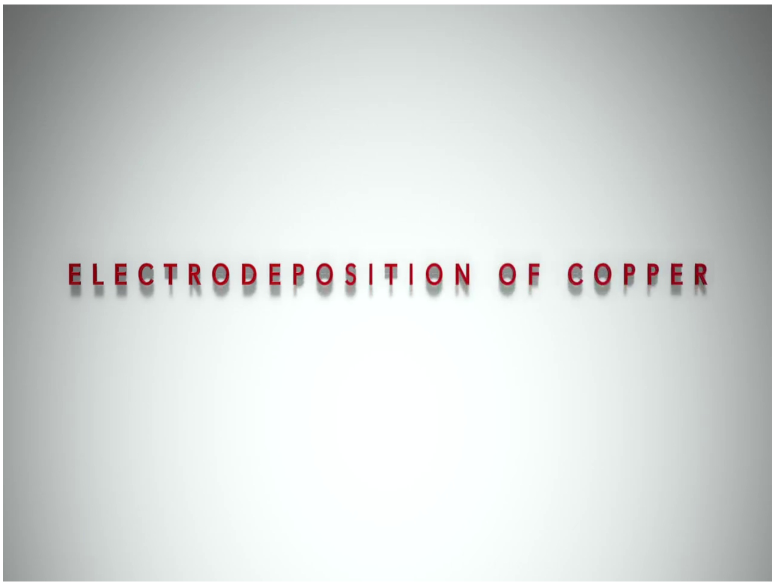

Hello students. Now, I will demonstrate you about the electro deposition of copper. For this process we need the following chemicals. Copper sulphate which will act as a precursor for the electro deposition of copper then 0.1 mole H2SO4 that will act as the solvent.

Now, this particular copper sulphate we need to measure of about 10 millimolar concentration and then like by calculating from the molecular weight given here 159.6 and then we will measure it in the measuring balance and we will mix it in 0.1 molar H2SO4 solvent.

After doing the measurement and preparing the solution of 10 millimolar of copper sulphate in 0.1 molar H2SO4 solution it will look like this of light blue colour. And the solvent is here type 1 di water. Now, after the preparation of this 10 millimolar of copper sulphate solution in

0.1 molar H2SO4 we will take it as the electrolytic solution for the electro deposition of copper process.

And now here we can see that we have working electrode, counter electrode and reference electrode and this is the electrochemical cell. By taking this measuring cylinder of 10 ml we will add this particular solution to this measuring cylinder and we will measure 10 ml and we will put it in the electrochemical cell for further analysis.

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So, after measurement of 10 ml of 10 millimolar copper sulphate solution in 0.1 molar H2SO4 now we will purge nitrogen into this solution to avoid the dissolved oxygen effect. So, here we can see this is a nitrogen cylinder from where the nitrogen will supply to this particular gun.

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So, we will set up this particular electrochemical cell with this gun. Now, on opening the knob the nitrogen bubbling will gradually start in the electrochemical cell which we can observe clearly that the dissolved oxygen is going out of the solution and the nitrogen will be purged to the solution. And then we will cover this particular electrochemical cell with aluminium foil to ensure that there is no reaction with environmental oxygen.

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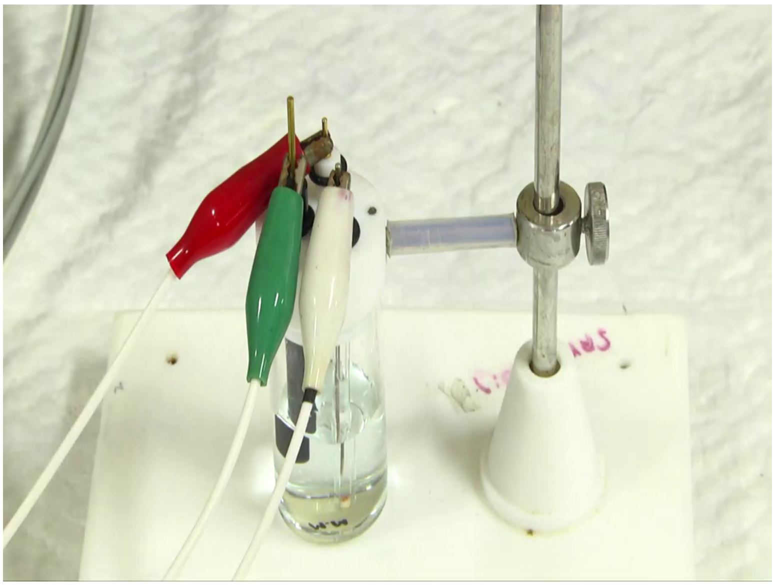

We will wait for 5 to 10 minutes and then we will do the further electro deposition process. So, now we will perform the electro deposition of copper here. So, by now you all know about the 3 electrode system where there is a working electrode, reference electrode and a counter electrode. These 3 are respectively attached by the crocodile clips to the CHI machine.

Now, this particular 3 electrodes are dipped in copper sulphate solution in H2SO4 medium. Now, I will perform the chrono-amperometric experiment and will show that how the copper gets reduced to copper from copper plus 2 to copper 0 in this working electrode region.

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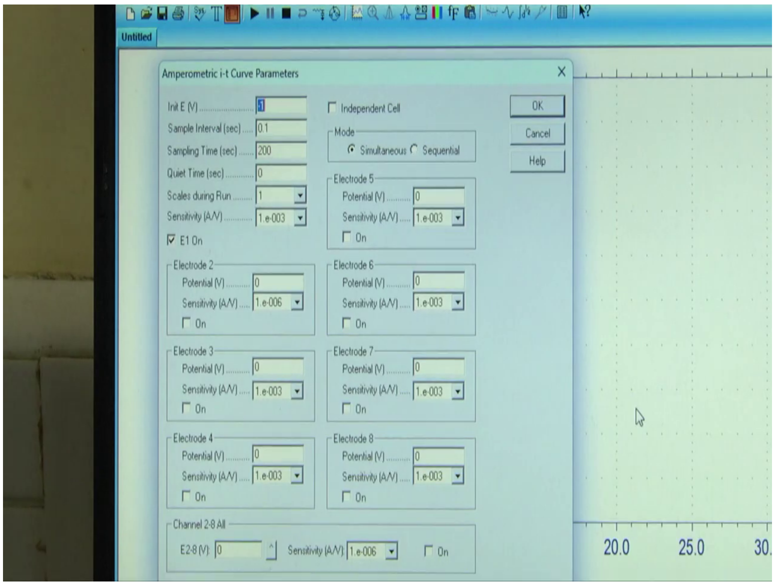

Now, we will set the parameters for the chrono-amperometric technique where the initial voltage we will put minus 1 volt, then the sampling interval for 0.1, the reduction time or the sampling time it will take 200 seconds that is for 200 seconds the copper will get reduced to copper 0. Then the sensitivity will be about 10 to the power of minus 3.

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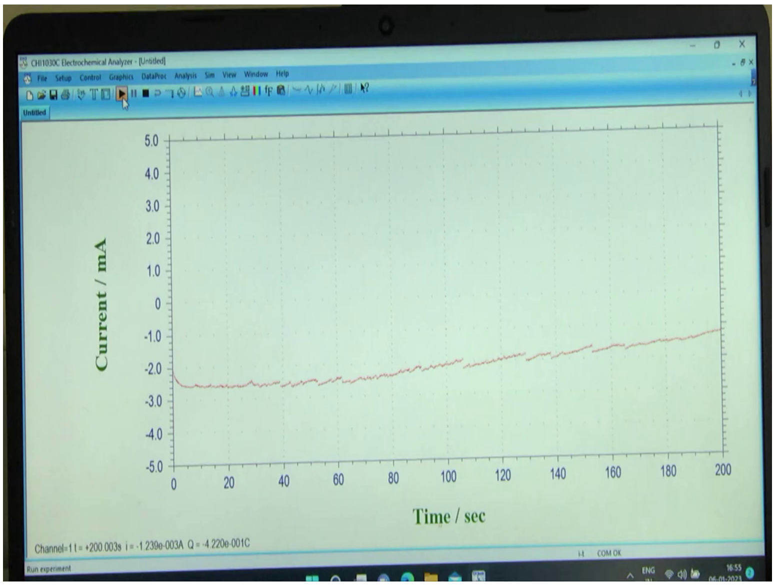

So, all parameters are set now. Now, we will run the experiment. So, the experiment will run for 200 seconds where the potential is constant minus 1 volt and we will see the current changes with time up to 200 seconds. So, in this period time period the particular copper 2 plus ions will diffuse towards the working electrode surface and will get reduced to copper 0 state and then we will see the color change after 200 seconds.

Now, we will wait for the time. Ok so, now this is the chrono-amperogram obtained. Now, we will observe the color change in the working electrode surface.

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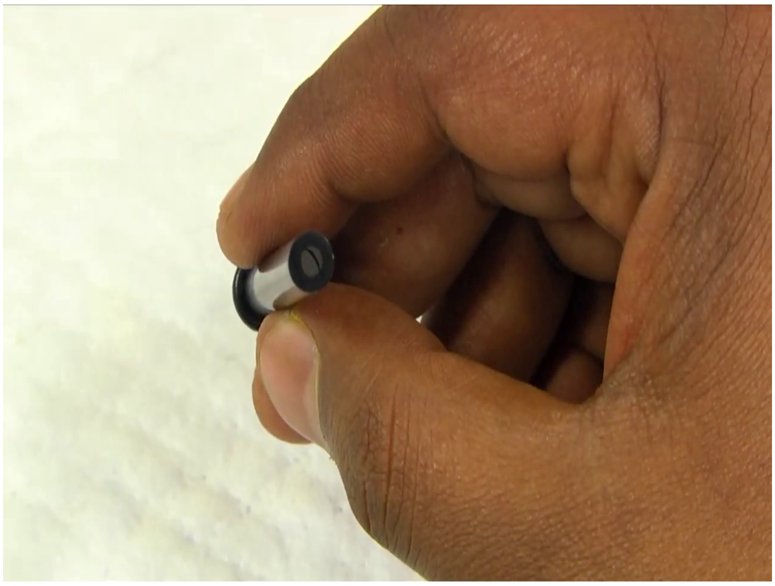

Now, for comparison purpose we will take a new glassy carbon electrode to compare the surface. Now, you can see that these are shiny surface here and we will compare this with our electro-deposited copper working electrode.

Now, we will see the changes in the working electrode surface. Now, we can see clearly the color change to reddish brown that is the characteristic color of copper nanoparticles, which actually absorbs the visible region light of bluish range and then reflects the reddish range color.

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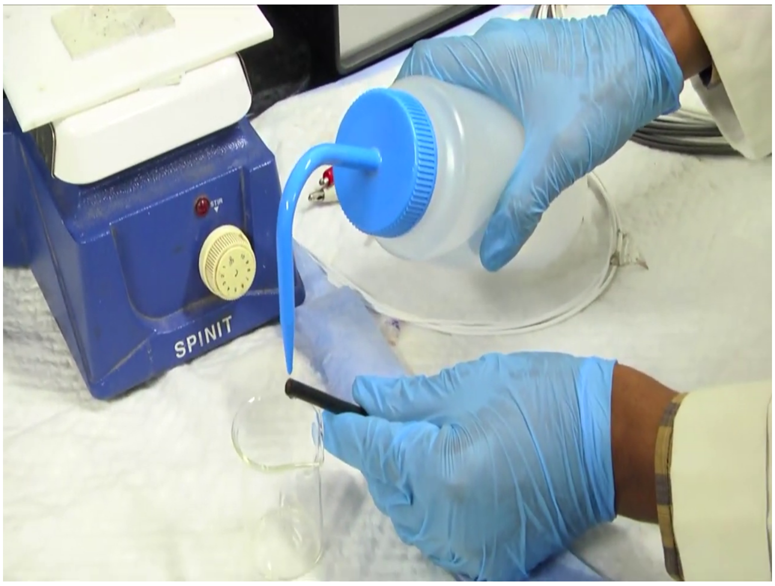

Now, to ensure that this particular copper nanoparticles are strongly bounded to the working electrode surface we will see that after washing with di type 1 water gently we will see the color has not changed.

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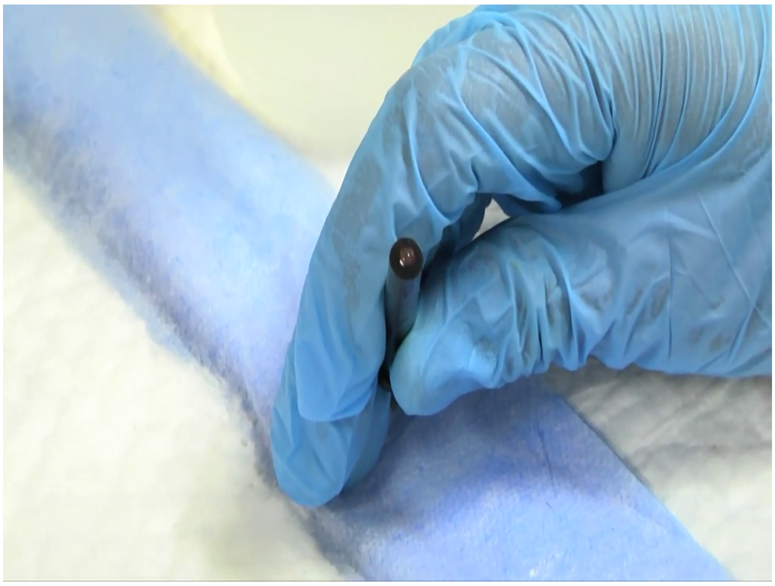

From this we can conclude that the electro-deposited copper after reduction from copper 2 plus to copper 0 state it is strongly bounded to the working electrode surface. So, the electro-deposition of copper we have already shown. Similarly, we can prepare by wet chemical synthesis copper nanoparticles by using reducing agents like sodium borohydride and stabilizers like glycerol and then we will can prepare the copper nanoparticles in powder form.

Then for further analysis we can drop cast those copper nanoparticles on the working electrode surface and we can then apply for different sensing applications.

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Hello everyone today I Jai Shukla will be talking to you about potentiostat. Now, before I start, I will first demonstrate newer device which is palmsens. It is a portable potentiostat that can be used for doing remote sensing at different locations and going to a locality where you cannot have all the experimental setup especially the ones which are quite heavy and quite bulky.

Now, let us first look into different aspects of this potentiostat. The first thing about this potentiostat is that it is portable which implies that it has a battery it does have a Bluetooth connection and our mobile apps can be connected to it. Now, with this application we can do electro chemical experiments by putting in our analyte.

Let us say if you are going into some village and trying to do some sensing for that village then we can take the samples on real time prepare them there itself and using this device we can do the experiments. So, let us see how it is being done.

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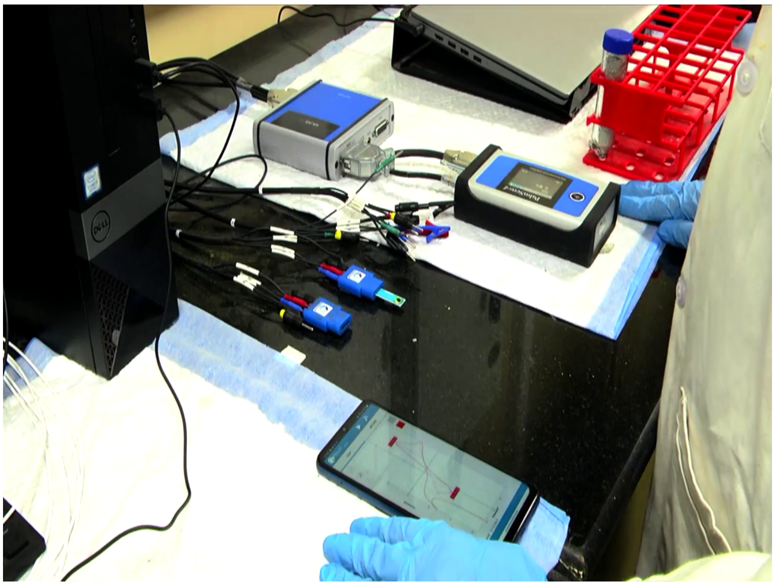

So, in order to do an electro chemical experiment using this potentiostat you can see it is right now not connected to any battery it has its own battery. This particular module is a multiplexing module, which allows us to do different electrode sensing consecutively. Now, we have attached a drop sense electrode onto the adapter of this potentiostat, which is connected through a wire. Now, I have put ferric ferrocyanide solution 5 millimolar onto the solution and I am ready to do the experiment.

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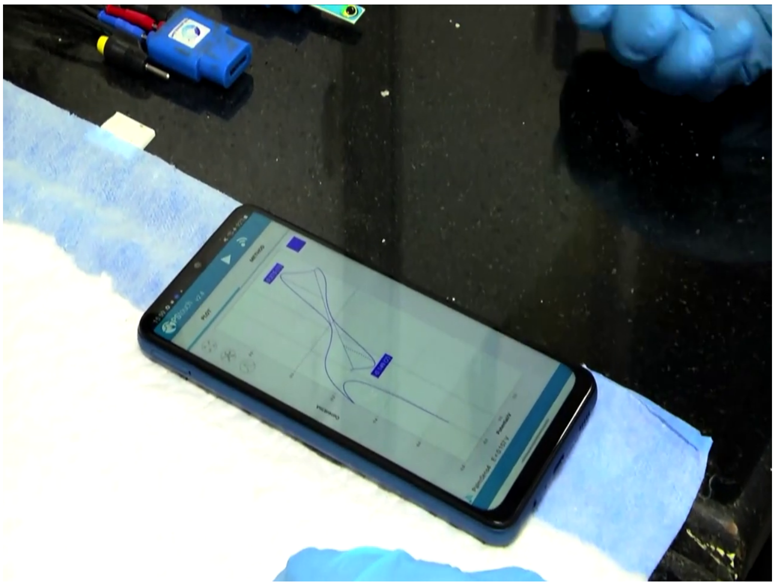

To do this experiment I have using this PS touch mobile app which is being provided by palmsens and after setting it this up I can just run the experiments and you can see you can see the experiment starts running. The values which are recorded from the sensors are being showed on the display and then I can send this value for further processing.

Now, we have seen how this potentiostat can be used for taking a diagnostic experiment or doing diagnostics at point of care. Now, these setups can help us reach a commercial goal of providing users or let us say if we want to we have developed some innovation we want people to use that innovation for their own self then we need to provide them such potentiostat.

Coming back these sort of potentiostat can be developed you by providing an electronic module. In this particular electronic module there are circuits, which generates a voltage wave form. The second circuit is which sends the current and finally, a module which can takes in the current values and the voltage values plot them together and send it to the analysis (Refer Time: 20:52), analysis device.

This analysis device could be a mobile phone it could be a desktop or it could be a IoT server which is bought by the user. In order to build these devices, the electronic components mentioned can be made by sourcing different parts. For example, let us say we want to measure a voltage between two electrodes then we can source a volt voltage measurement module.

For current we can similarly source a current measurement module also we can also design our own circuits using op amps, resistors and capacitors at the same time to get the required analysis. These circuits are active in nature, but these needs to be controlled. For that you need to add another module which is a microcontroller.

Now, this microcontroller can be programmed using an Arduino system or it can be programmed using bare metal programming strategies which are generally writing a C code for your particular microcontroller which can do the specific experiment.

Apart from that there are different setups for example, you can use a source meter, you can use a power supply oscilloscope setup along with a multimeter to do this sort of experiments which we will see shortly. To develop the mobile application for this potentiostat you would need the knowledge about some application development which can be let us say native mobile application development let us say if we want to talk about android and swift for iOS then it could also be done using some frameworks like angular, ionic or native scripts which are readily available.

Flutter and flutter using dart is another option that you can use to build this device, build the software that can run on android or iOS devices. One such device that is being developed inside our lab is called NBS analyzer. Now, we have this strategy which I have mentioned to you earlier we have used that to build this device. First let me show you the mobile app that we have developed for this one.

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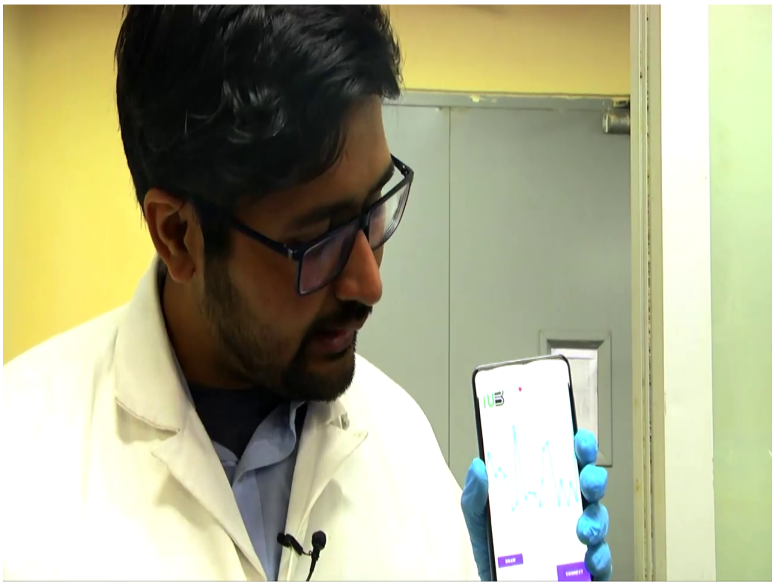

This is the NBS analyzer mobile app that we have developed. Now, in order to develop this particular application, we have used a native android studio and based on that we have created a Bluetooth connection, a graph element and a connection element that connects to our device.

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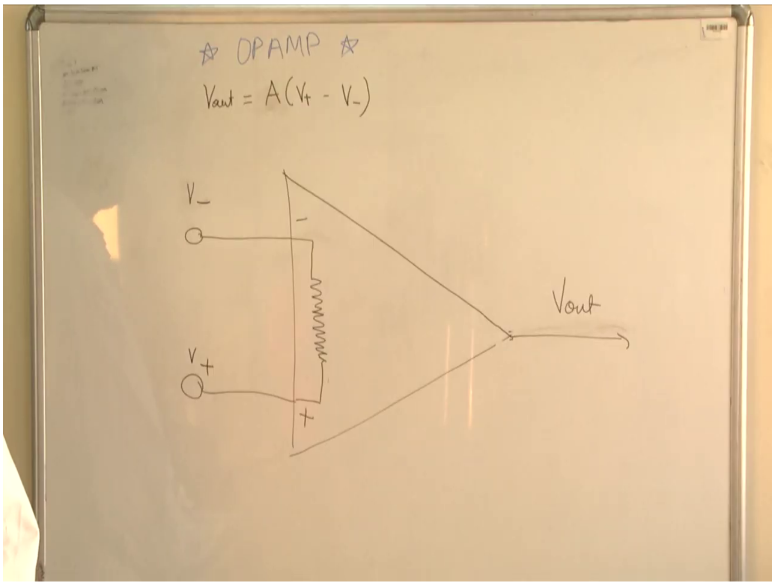

To build a potentiostat one of the very important component is a op amp. It has three terminal non-inverting terminal, inverting terminal and V out. Now, these three terminals are connected in such a way that when you apply a potential difference between V plus and V minus it is being multiplied amplified by a factor of A and that comes out at the V out.

The property of this op amp is that the terminal between the non-inverting terminal and the non-inverting terminal are connected with a very high resistance. So, let us say if I only apply some voltage at the non-inverting terminal that voltage will again be reflected at the non-inverting at the inverting terminal because since this resistance is very high. So, there would not be any current there.

So, making the inverting terminal a virtual voltage with respect to the non-inverting terminal. The current values inside electrochemical cells can be satisfied with the configuration of the

V out terminal. So, let us say if there is any need of and current value that can flow through the V out terminal of this particular op amp. Now, we will see how we can use these op amps to build a potentiostat.

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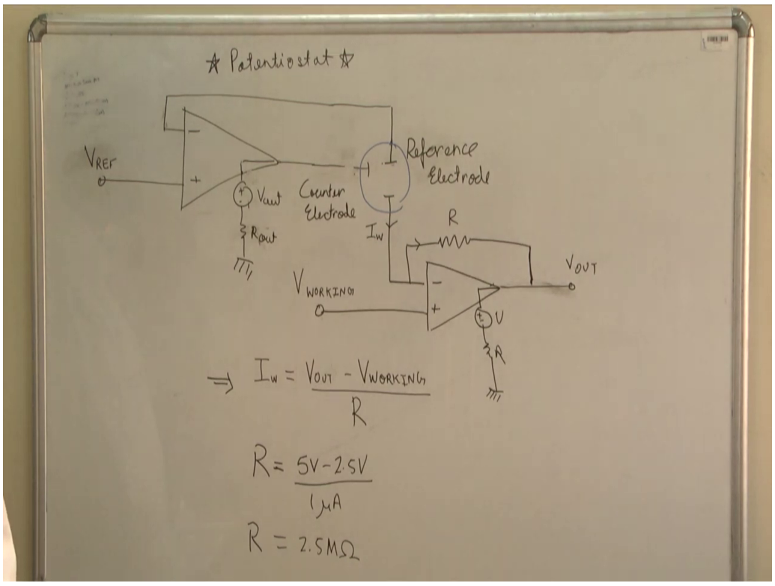

Now, we will see how we can use op amps to build a potentiostat. The first op amp that you see here has V reference applied to the non-inverting terminal. This voltage is then reflected at the inverting terminal of the op amp because as we discussed earlier this adds a virtual potential for this because there is a very high resistance.

Now, the non- the inverting terminal is connected to the reference electrode. This way whatever voltage we apply at the non-inverting terminal become becomes the voltage of the reference electrode. This also from the previous discussion we also know that the current through the reference electrode should be minimal. As you can see the particular circuit has a very high resistance in it path.

So, the current through the reference electrode is limited by the electronics as well. Now, the counter electrode is connected to the V out of the potentiostat. This means that the V out at the moment can be any value that can help us satisfy the required working potential, working current ok I will just repeat this line.

Now, the counter electrode is connected to the V out of the op amp. This means that current counter electrode can now provide with the features of op amp the required current which is required to make the working electrode work.

Now, for the working electrode this op amp as like the voltage set a for its electrode. As you can see the non-inverting terminal is connected to the V working and that will be then seen at the inverting terminal. Hence the working electrode will be set at the V working voltage. Now, in order to sense the current through the working electrode what we have is a connection between non-inverting terminal and the V out.

Now, since we already know the current which flows through the inverting terminal and the non-inverting terminal is negligible. So, we can assume that the current through the working electrode will be flowing through this particular path. Now, the hence this gives out the following equation. In order to make this picture more clear let me tell you one more fact.

The inside of an op amp can be modeled like this. Which basically means that the output terminal of the op amp is controlled is a voltage control current's voltage source and has a very small resistance connected to it. Now, let us say there is any requirement of current which is needed to make the counter and the working electrode work V out can adjust (Refer Time: 28:15) to provide that particular current because that will be the limiting case for the electrochemical setup.

Then in this particular setup we can see the current which is flowing through the working electrode then can go to V out and then finally, can be drained out. This way we will be able to see the effect of current of the working electrode as a form of V out. From this particular equation we can set the value of R depending upon the maximum current that we want to observe.

Let us say for R op amp the maximum potential that it can measure is 5 volt and let us say the working potential that we are going to set the maximum value is 2.5 volt and the maximum current that we want to see is 1 microampere then we can use the following equation to find the R value.

That will be 2.5 mega ohms for this particular circuit. Now, this is a oversimplified model of a potentiostat, but it can help you understand how this particular device work. Now, you can see in order to perform any experiment let us say cyclic voltammetry, differential pulse voltammetry, chronoamperometry or any other experiment we can use this particular setup.

The good thing about this particular setup is that we can set V ref to any potential let us say 2.5 and then we can vary the working potential to get the effect of minus 1 and plus 1 voltage with respect to the reference electrode which we generally see for an electrochemical setup.

We know that any voltage between 0 or power supply or let us say if any negative power supply is present in the circuit can be used for setting this potential. Hence even only with this positive power supply we will still be able to observe negative voltages and their effects on the working electrode with respect to the reference electrode.

Now, this particular circuit can be connected to a microcontroller that microcontroller can set the V reference and can measure V out with the help of ADC. Then we will have a programmable a potentiostat setup. Now, the good thing about microcontroller these days these already comes with a lot of modules which helps you connect through Wi-Fi, Bluetooth etcetera.

So, by making the by writing the correct code for the microcontroller you will be able to make your own potentiostat. The few example of microcontroller that you can use for your circuit is

ESP32, Arduino, Arduino Mega or Beagle boot, Raspberry Pi, Raspberry Pi Beagle etcetera. All of these come with the necessary feature for you to make this particular circuit work.

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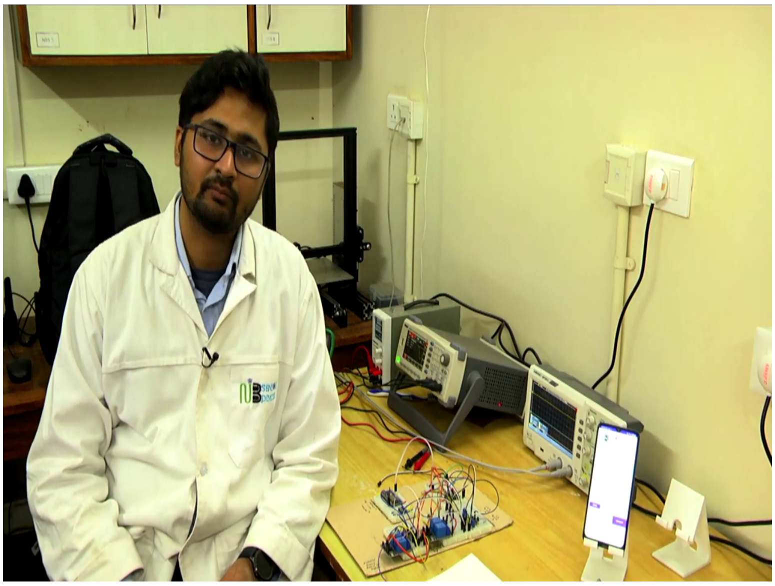

Now, let us get into the practical aspect of building a potentiostat. So, as we have previously discussed that using op amps and resistors and microcontroller, we can build a build such system. Now, to test them we need certain devices. Now, few of them are oscilloscope which can help you verify whether the signal which you are trying to generate or the voltage waveform which you are trying to generate is actually there.

You can connect the oscilloscope towards the output pin that you want to observe and then you can see the voltage output. There is function generator which can help you take the error value from the generated functions from the generated waveform from your devices and then can help you analyze whether the device components is working correctly or not. There is another important aspect is that you can also test the current output module and the current sensing module using these two setups.

Just for current sensing what you have to do is that you have to add a very small resistance in the series to the connection line where you want to observe the current. The third thing is the power supply. Power supply basically provides the required voltages for operating your device. Now, let us say in this case I am using a ESP32 module. So, it requires a power voltage of 5 volt and since it is a development volt it has a 3.3 voltage output and 5 voltage output. So, that can be then supplied using the power supply.

To verify the connection, I use my USB connection to the computer and that connection sends me the serial output and based on those particular serial outputs I check whether my potentiostat is working fine or not. The last thing is that after making all of this circuit on this setup this which is called a breadboard setup it has several pins and on that you can attach your components by just pinning them onto the breadboard.

This setup does allow you to prototype your circuit quickly, but they do not provide a very good noise cancellation. Also since there are lot of long wires and a lot of long connection inside this breadboard it results into a very noisy signal. So, if you expect your device to work device work on a breadboard setup it is slightly difficult. So, the final thing what you have to do is that after testing all your components whether they work on a work or not you have to convert them into a PCB.

For PCB you can use softwares like KiCad, Altium designer, Eagle to design your to design and layout your PCB with different components that you have just tested. Finally, the final outcome of this whole project is a device that can help you do your experiments or help you make a commercial commercially viable output that can be used at let us say remote places in hospital or for personal care.

For that you need you also need to make a mobile app and the respective firmware for that. It has to it has to work with Bluetooth; it needs to work with Wi-Fi. Those modules you can program in those particular softwares. The particular software are in few cases is code link or you can also use Java to make your mobile app. But there are new upcoming frameworks which simplifies these development using Angular Native, React Native or Ionic, Expo etcetera.

These particular frameworks help you make your product at the end and helps you do your experiments at in a peaceful way and so, that you can get your results. This particular potentiostat has several other blue blocks and you can see these are quite bulky. These are nothing but the switches which are electrically controlled using a microcontroller.

These are called delays. These are also very important component when you want to change your system from one setup to other setup by having physical connection. The other important component about this particular setup is that the microcontroller. Microcontroller can if you search them online you will find that these microcontroller are very small ICs, but in order to make them work you need to have external peripherals which include clock which include ADC different power supply module.

So, in order to avoid those complication in your project what you can do is that you can buy development boards, which already comes with all the necessary power supply modules, USB connection order, serial communication modules and etcetera. That will help you make your project come alive in very small amount of time.

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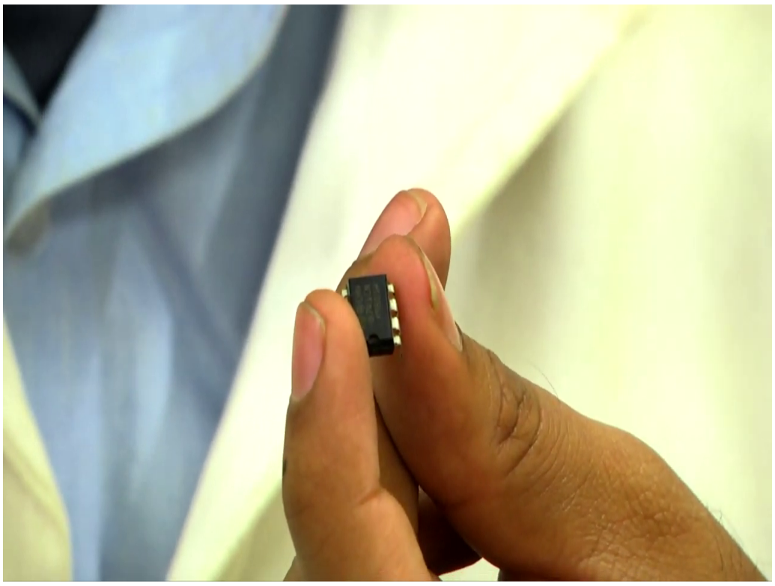

The component which I have at my hand is an IC. It is an Integrated Circuit that contains the logical part of your circuits. Now, these integrated circuits have different smaller components designed into them which helps you reach your purpose. As we discussed previously, op amps now, how these come in the real world? These come in this particular form of packages.

These are also integrated circuits which helps you achieve your purpose. Similarly, current modules, voltage module and different Bluetooth modules also come in more or less similar packages. This can also be very much smaller than this. They are called surface mounted devices called SMDs and they can improve in making your device smaller.