# Synthesis and Characterization of a Novel Nanomaterial-Strontium Ruthenate(SrRuO$_3$) Design for Supercapacitor

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**Abstract:**We have under taken the synthesis and characterization of a novel nanomaterial Strontium Ruthenate and explored the possibility of its role in design of a supercapacitor. Strontium Ruthenate is synthesized by solution route method and it is characterized by TGA, XRD and SEM-EDXA. Further it is designed as an oxide electrode to determine its I-V characteristics in different electrolyte solutions. Specific capacitance values obtained are so high, which reviled its supercapacitor properties.

## 1. Introduction

Now a day the demand of electrical energy is increasing. But it faces one drawback that is it's relatively difficult to store it quickly. Batteries can hold large amounts of power, but they take hours to charge up. Capacitors, on the other hand, charge almost instantly but store only tiny amounts of power. In our electric-powered future, when we need to store and release large amounts of electricity very quickly, it's quite likely we'll turn to supercapacitors but before that we have to know what are these and how they works? Batteries and capacitors do a similar job that is storing of electricity but in completely different ways:

Batteries have two electrical terminals (electrodes) separated by a chemical substance called an electrolyte. When you switch on the power, chemical reactions happen involving both the electrodes and the electrolyte. These reactions convert the chemicals inside the battery into other substances, releasing electrical energy as they go. Once the chemicals have all been depleted, the reactions stop and the battery is flat. In a rechargeable battery, such as a lithium-ion power pack the reactions can happily run in both directions so we can usually charge and discharge hundreds of times before the battery needs replacing. But Capacitors use static electricity (electrostatics) rather than chemistry to store energy. Inside a capacitor, there are two conducting metal plates with an insulating material called a dielectric in between them, it's a dielectric sandwich. Positive and negative electrical charges build up on the plates and the separation between them, which prevents them coming into contact, is what stores the energy. Capacitors have many advantages over batteries: they weigh less, generally don't contain harmful chemicals or toxic metals, and they can be charged and discharged billions of times without ever wearing out. But they have a big drawback too: their basic design prevents them from storing anything like the same amount of electrical energy as batteries. So to increase the capacitance we can have, either by using a better material for the dielectric or by using bigger metal plates. Like an ordinary capacitor, a supercapacitor has two plates that are separated by a dielectric. The plates are made not from metal but from a porous substance such as powdery, activated charcoal, which effectively gives them a bigger area for storing much more charge. Like a battery, a supercapacitor has an electrolyte (electrically active chemical) inside it, separating its plates, which is more like the electrolyte in a battery than the dielectric in a conventional capacitor (which might just be a piece of plastic or even air). In a conventional capacitor, positive charges form on one plate and negative charges on the other with the dielectric sitting in between them, keeping the charges safely apart. In a supercapacitor (1,2,3,4) the electrolyte is electrically active and adds another dimension: the charged plates polarize the electrolyte, making positive ions inside it move one way and negative ions the other, and causing a second set of charges to form. This makes what's called an electric

double-layer that allows the plates to store more charge. (This, incidentally, is why supercapacitors are often referred to as double-layer capacitors, also called electric double-layer capacitors or EDLCs). Unlike in a battery, the positive and negative charges in a supercapacitor are produced entirely by static electricity; no chemical reactions are involved.

A different kind of capacitance can arise at electrodes of certain kinds, for example metal oxide, when the extent of faradaically admitted charge depends linearly, or approximately linearly, on the applied voltage. For such a situation, the electrode behavior is equivalent to, and measurable as, a capacitance. This capacitance can be large but it is faradaic and not electrostatic (that is non-faradaic) in origin. This is hence an important difference from the nature of double-layer capacitance, so it is called "pseudocapacitance". This kind of pseudocapacitance (5,6) can originate when an electrochemical charge-transfer process takes place to an extent limited by a finite quantity of reagent or of available surface. Several examples of pseudocapacitance can arise, but the capacitance function is usually not constant and, in fact, is usually appreciably dependent on potential or state of charge.

In this piece of work we have synthesized the nanomaterial strontium ruthenate and observed the I-V characteristic which shows that it is a good material for a supercapacitor.
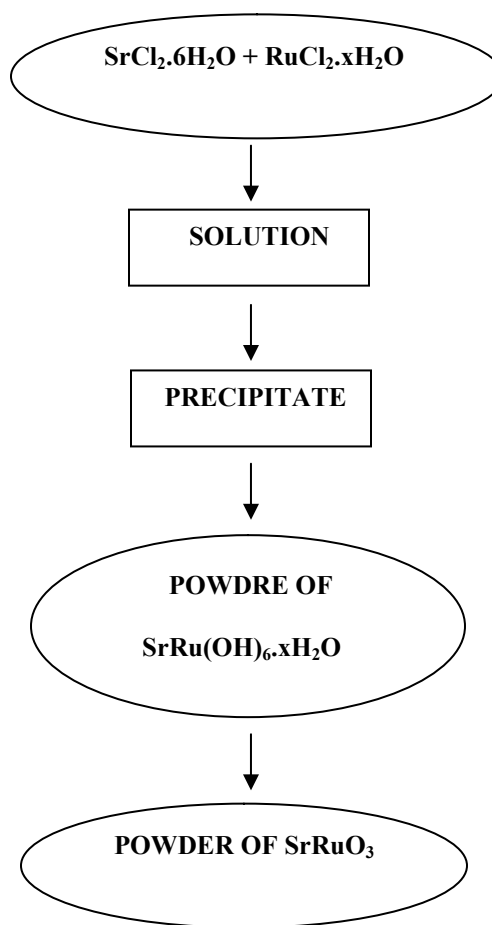
## 2. Experimental

The experimental work has been done in Centre For Materials for Electronics Technology(C-MET),Pune.

### Synthesis of Nanomaterial Strontium Ruthenate (SrRuO$_3$):

The solution synthesized nano-powders of strontium ruthenate were characterized by the following instruments. Thermal stability of ruthenate powder was investigated by using thermal analyzer (Mettler, Toledo, Model 851e) in dynamic nitrogen atmosphere in the temperature range 25 $^o$C to 800 $^o$C. Surface morphology, particle size and elemental composition were determined by using Scanning Electron Microscope-Energy Dispersive X-ray Analyser SEM-EDX, Model XL 30, Philips, Holland). X-ray diffractograms (XRD) were recorded on X-ray diffractometer (X-ray Generator, Model PW 1729, Philips, Holland) by using CuKa (k =1.5405 A°) and Ni as the filter. Stoichiometric amount of SrCl$_2$.6H$_2$O and RuCl$_3$.xH$_2$O are dissolved separately in 0.04% (w/w; 1.0 mM) Cetyl Trimethyl ammonium bromide (CTAB) solution as surfactant. The two solutions were mixed, stirred and the pH of the

mixture was slowly increased up to 9.41 by addition of dilute NaOH.

$$\boxed{SrCl_2.6H_2O + RuCl_2.xH_2O}$$

$$\downarrow$$

$$\boxed{SOLUTION}$$

$$\downarrow$$

$$\boxed{PRECIPITATE}$$

$$\downarrow$$

$$\boxed{POWDRE\ OF\ \ SrRu(OH)_6.xH_2O}$$

$$\downarrow$$

$$\boxed{POWDER\ OF\ SrRuO_3}$$

**Flow Chart – 1**

The reaction mixture was maintained at pH between 9.41 by using dilute HCl and NaOH solutions for about 1 h by constant stirring.The reaction mixture was allowed to settle for an hour, filtered and washed with distilled water and the powder was dried at 100$^o$C. The dried sample was ground to fine powder. This powder was further dried in vacuum oven at 250 $^o$C for 4 h. The synthesis is shown in the flow chart flow chart-1.

## 3. Results and Discussion:

Thermal stability i.e. weight loss/original with different temperatures of the prepared samples were investigated by using Thermal Analyzer (Mettler Toledo 851e) in dynamic nitrogen atmosphere in the temperature range 30°C to 900°C at 10°C/min. The surface morphology, particle size

and elemental composition were determined by using Scanning Electron Microscope with Energy Dispersive X-Ray Analysis (SEM-EDXA, Model XL 30, Philips, Holland Phase identification was carried out by X-ray Diffractometer (X-Ray generator, Regaku Miniflex, Japan) employing a scanning rate of 2°/minute in diffraction angle 2θ range from 10° to 80° and CuKα radiation (λ=1.5405 A°).

### 3.1 Thermal Analysis(TGA/DTA):

Thermal analysis of strontium ruthenate synthesized by solution route method as shown in the Fig.2. The first weight loss is due to water elimination and the second weight loss is due to the formation of Sr and Ru complex hydroxides. The third weight loss is due the formation of $SrRuO_3$.
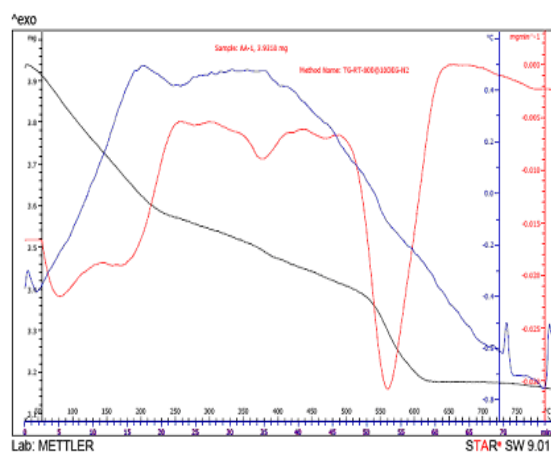


Fig.2:TGA/DTA result of nanomaterial $SrRuO_3$

### 3.2 X-Ray Diffraction Studies:

Fig.3 is the XRD of strontium ruthenate (SRO) prepared at pH 9.41 by solution route and dried at 250 °C in air. According to the theoretical and experimental values of $SrRuO_3$ the majority of the peaks matches with the reported strontium ruthenate. The three XRD major peaks of $SrRuO_3$ which are matching with the $RuO_2$ peaks and the above XRD pattern matched with JCPDS data (JCPDS No. 43-1027).
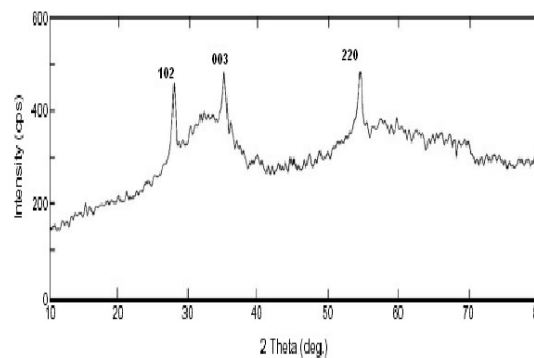


Fig 3:XRD result of nanomaterial SrRuO3

### 3.3 Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis(SEM-EDXA):

The scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDXA) for prepared materials are explained here. Figure 4 shows the SEM image of nanocmaterial $SrRuO_3$ synthesized by solution route method. This SEM image clearly shows the morphology of $SrRuO_3$ nanoparticles with an average particle size of 50-100 nm. Figure 5 is the EDX spectrum of nanomaterial $SrRuO_3$. Table 1 gives the element composition of $SrRuO_3$ which matched with standard reported value.
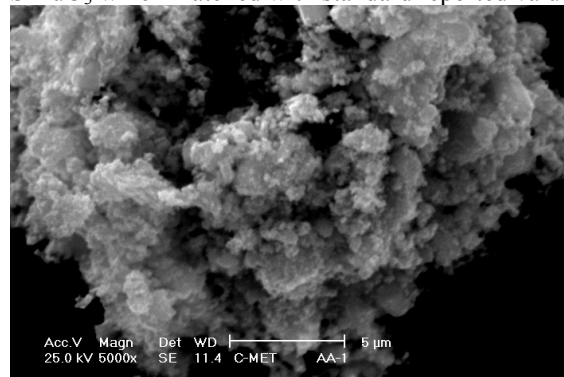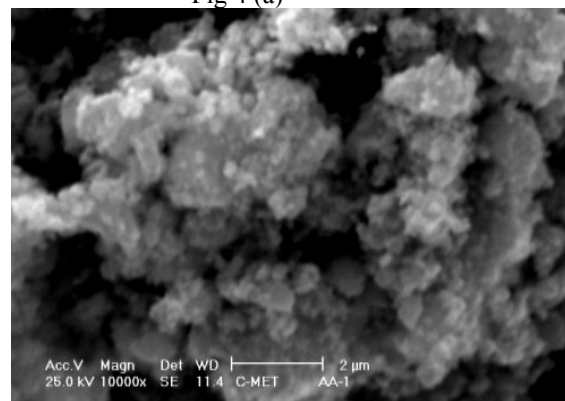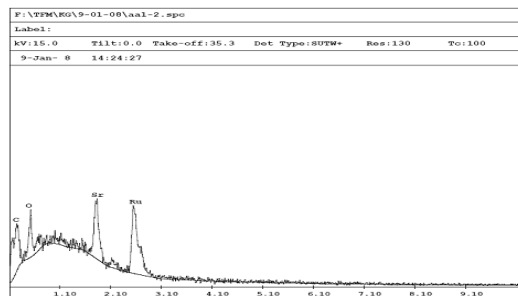


Fig 4 (a)



Fig 4 (b)

Fig. 4. SEM micrographs of a) nanosized $SrRuO_3$ (× 4000), b) nanosized $SrRuO_3$ (×8000).

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kV:15.0    Tilt:0.0  Take-off:35.3   Det Type:SUTW+   Res:130    To:100
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Fig 5: EDX spectra of $SrRuO_3$

Table 1. The element composition of $SrRuO_3$ which matched with standard reported value



EDAX ZAF Quantification (Standardless)
Element Normalized

| Element | Wt % | At % | K-Ratio | Z | A | F |
|---|---|---|---|---|---|---|
| C K | 29.92 | 59.09 | 0.1055 | 1.1338 | 0.3110 | 1.0001 |
| O K | 19.09 | 28.31 | 0.0383 | 1.1118 | 0.1805 | 1.0001 |
| SrL | 17.68 | 4.79 | 0.1464 | 0.8345 | 0.9850 | 1.0070 |
| RuL | 33.31 | 7.82 | 0.2701 | 0.8393 | 0.9663 | 1.0000 |
| Total | 100.00 | 100.00 | | | | |

| Element | Net Inte. | Bkgd Inte. | Inte. Error | P/B |
|---|---|---|---|---|
| C K | 26.59 | 9.35 | 3.80 | 2.84 |
| O K | 21.66 | 17.09 | 4.84 | 1.27 |
| SrL | 43.74 | 25.26 | 3.20 | 1.73 |
| RuL | 56.41 | 12.41 | 2.48 | 4.54 |

## 4. Electrode Preparation

Electrodes were prepared by painting slurry of the powder mixed with a solution of water and Triton X-100 (10:1) on nickel foils. These foils were previously etched in boiling HCl to remove oxide layers and to roughen the surface. The foils were point welded to a nickel wire as a current lead. To attach the coating and to burn out the binder the electrodes were annealed at 300–400°C for 2h in air after painting. The electrodes were 15×15 mm2 and coated on one side only. Typical amounts of active mass were in the range of 2 mg cm-2, which was determined by difference weighing before and after the coating. There was no detectable weight change of uncoated nickel foils due to surface oxidation of the nickel foil after such an annealing procedure.

## 5. Characteristic I-V Curve

A three electrode arrangement in a beaker type class cell was used to determine the electrochemical behaviour. The counter electrode consisted of a platinum sheet. Hg/HgO was used as the reference electrode in the same electrolyte. The electrolyte was 6M KOH. Cyclic voltammograms at various voltage sweep rates were recorded using an EG&G PAR model 273 potentiostat, controlled by a microcomputer. Specific capacitances were calculated by integrating the current of a CV half cycle and relating the charge to the voltage range and

the weight of the active mass. Because the mass utilization of the porous active layer is very good the current densities in the CV diagrams are given with respect to the active mass loading rather than to the geometric area. The specific capacitance of 8 F g$^{-1}$ is produced from the I-V plot using the formula: C = I/(S x M)  where I is average current, S is scan rate and M is mass of the material coated over your electrode.
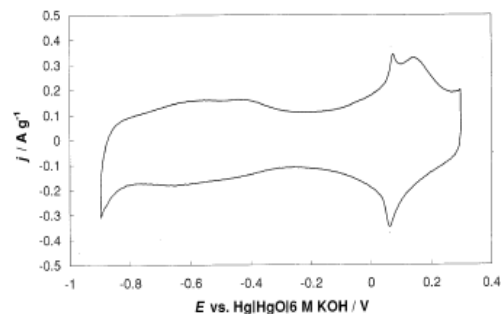


Fig 6: CV taken at 20 mV s-1 of co precipitated $SrRuO_3$

## 6. Conclusion

We successfully synthesized Strontium Ruthenate nanomaterial at room temperature and pressure. The formation of pure phase of $SrRuO_3$ was confirmed by XRD.  TGA/DTA is used to analyse the material and stepwise weight loss was correlated with formation of various intermediates. SEM images clearly showed the morphology of $SrRuO_3$ nanoparticles with an average particle size of 50-100 nm. I-V characterization was used for calculation of specific capacitance of the material. A high capacitance 8 F/g was obtained using $SrRuO_3$ coated electrode. Materials showed enormous potential for use as an active material for super capacitors.

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