

POLYMER COMPOSITE REDUCTION WITH ZnO CATALYST

Submitted in partial fulfillment of the requirements for the award of

Bachelor of Technology degree in
Chemical Engineering

By

VISHAL B (38190015)



**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with grade "A" by NAAC

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI – 600119

MAY 2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **VISHAL B** (38190015) who have done the project as a team, who carried out the project entitled "**POLYMER COMPOSITE REDUCTION WITH ZnO CATALYST**" under our super vision from

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DECLARATION

I **VISHAL B(38190015)** here by declare that the Project Report entitled “**POLYMER COMPOSITE REDUCTION WITH ZnO CATALYST**” done by me under the guidance of **Dr. S. SATHISH Ph.D.** is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

.

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ABSTRACT

Polymer composites are materials exhibiting modified physical and chemical properties and are also found to show enhanced catalytic activity. Recently, different types of polymer composites of high catalytic activity have been prepared by incorporating nanomaterials such as Zinc oxide (ZnO), Titanium dioxide (TiO₂), graphene, activated carbon etc. Polymer composites of incorporated nanomaterials were synthesized by different self-propagation techniques. To further add on to the existing list of polymer composites, we have synthesized a new polymer composite of PVA with incorporated metal and ZnO nanomaterial. The synthesized composite has been characterized by UV-visible spectroscopy, FT-IR and SEM-EDAX. The optical properties of the synthesized composite were studied using UV-DRS. The FT-IR spectroscopic analysis were carried out to confirm the structural modification in polymer composite. SEM-EDAX reveal the surface morphology and elemental composition of the synthesized composite. The thermal stability of the prepared polymer composite is revealed by TG analysis. This paper further studies the ability of the synthesized polymer composite to reduce hexavalent chromium and has shown improved efficiency.

Keyword : morphological analysis, polymer degradation, PVA, Chromium reduction

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LIST OF ABBREVIATIONS

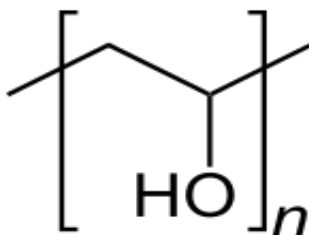
NPs	-	Nanoparticles
PVA	-	Polyvinyl Alcohol
Cu	-	Copper
ZnO	-	Zinc Oxide
Wt	-	weight
Cr	-	Chromium
CuSO ₄	-	Copper Sulphate
ml	-	Millilitre
mm	-	millimeter
FTIR	-	Fourier Transfer Infrared Radiation
SEM	-	Scanning Electron Machine
UV-vis DRS	-	Ultra-violet visible diffuse reflectance spectroscopy
TGA	-	Thermogravimeter Analysis
EDAX	-	Energy dispersive X-Ray Analysis.
G	-	Gram
Fig	-	Figure
HCl	-	Hydrochloric Acid

CHAPTER 1

INTRODUCTION

The polymer degradation is the reduction in the physical properties of a polymer, such as the strength, caused by changes in its chemical composition. Polymer and particularly plastics are subjected to degradation at all stages of their product life cycle, including their production, use, disposal into the environment and recycling.

PVA is a water soluble synthetic polymer. It has the idealized formula $[\text{CH}_2\text{CH}(\text{OH})]$. It is used in papermaking, textile warp sizing, as a thickener and emulsion stabilizer in PVA adhesive formulations, in a variety of coating. It is colourless and odorless (Hallensleben ML 2000). It is commonly supplied as beads or as solutions in water without an externally added crosslinking agent, PVA solution can be gelled through repeated freezing-thawing, yielding highly strong. PVA structure.



PVA composite is a synthetic polymer which possesses hydroxyl groups and is, thus, highly soluble in water. By adding solubility properties, PVA is biocompatible. It is also due to its hydroxyl groups, which can be chemically modified since PVA allows the insertion of other polymer or functional groups to increment its use

1.1 PROPERTIES

CHEMICAL FORMULA	:	$\text{C}_2\text{H}_4\text{O}$
DENSITY	:	1.19–1.31 g/cm ³
MELTING POINT	:	200°C
REFRACTION INDEX	:	1.477@ 632nm

Polyvinyl alcohol has excellent film-forming, emulsifying, and adhesive properties. It is also resistant to oil, grease, and solvents. It has a high tensile strength and flexibility, as well as high oxygen and aroma barrier properties. However, these properties are humidity dependent: water absorbed at higher humidity levels acts as a plasticiser, reducing the polymer's tensile strength while increasing its elongation and tear strength.

1.2 POLY VINYL ALCOHOL:

PVA is not made by polymerizing the equivalent monomer, vinyl alcohol, because this monomer is thermodynamically unstable when tautomerized to acetaldehyde. Instead of acetate, PVA is made by hydrolyzing polyvinyl acetate or other vinyl ester-derived polymers having formate or chloroacetate groups. Typically, base-catalyzed transesterification with ethanol is used to convert polyvinyl ester

1.3 PVA COMPOSITE WITH METAL (CuSO_4)

Pva composite with metal CuSO_4 Freshly prepared 5 mol% PVA and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ solutions were taken in proportion ($x = 1, 2, 3, 4$, and 5). mol% copper content for the preparation of 5 mol% PVA: x mol% Cu^{2+} polymer films. For around 10–20 minutes, these solutions were vigorously mixed and evenly distributed over the surface a plate made of glass. The films were re-moved from the glass plate after 48 hours of drying, in the 30–350°C temperature range.



Fig : 1.1 REACTION OF PVA+ CuSO_4

After drying in heat oven for 48 hours PVA composite with metal its become crystal powder.

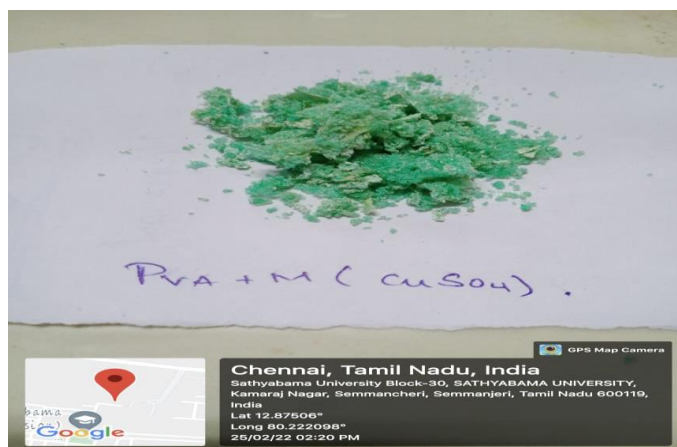


Fig : 1.2 SAMPLE OF PVA(METAL)

1.4 PVA METAL WITH CATALYST

PVA metal composite with ZnO catalyst. For the synthesis of nanomaterials, the sol-gel and combustion/self-propagation methods are reported to be cost-effective, simple, and harmless. The aqueous and nonaqueous sol-gel procedures are the two classifications of the sol-gel techniques. Whereas, the condensed/solid phase and solution phase synthesis approaches are the two classifications of self-propagation technique.

1.5 Preparation of PVA metal with ZnO

At first, aqueous 10 wt% PVA solutions were provided by dissolving 10 g of PVA in 100 ml deionized water and then refluxing at 80° C for 4 h. After that different amounts of ZnO nanoparticles (5,10,15,20 wt% of ZnO) were dispersed in 3.75 g de-ionized water and then sonicated for 2 h. for preparation of PVA/ZnO solution and adding up with CuSo4 for catalyst composite.

1.6 PVA COMPOSITE AS NANOCATALYST

The Nanocatalyst of PVA is ranging from 1 nm to 10 nm in size is called PVA nanocatalyst. It is used to reduction the PVA catalyst by ZnO particles. This nanoparticles are made to happen in reduction reaction through chromium hexavalent is to reduce the stability of polymer from hexavalent to trivalent from that its loses stability . Chromium is very poisonous solution. By polymer it is becomes less poisonous than before the reaction, these are several synthetic method to carry out PVA METAL nanoparticles through reduction process. This most common in them is wet chemistry.

Most of these reaction act as reduction agent. The most common shape of PVA metal nanoparticles is crystal in size. The synthesis of PVA composite in metal with ZnO particles.

Despite its numerous advantages, among increased adsorption capacity and others, the use of nanomaterials in AOPs is still in its early days leaving many unknowns regarding its toxicity to humans and the environment (Hillie and Hlophe 2007). The effects of fine nanoparticles ZnO powder on humans are still unknown.

An alternative strategy for reducing ZnO release or contact with the environment is to combining them with bonding clay to make small pellets nanoparticles ZnO composites was used in this experiment.

CHAPTER 2

LITERATURE SURVEY

Removal of Metaldehyde Through Photocatalytic Reactions Using Nano-Sized Zinc Oxide Composites(F. C. Doria & A. C. Borges & J. K. Kim & A. Nathan & J. C. Joo & L. C. Campos) . Influence of Zinc oxide of nanoparticles on structural and electrical properties of polyvinyl alcohol.(S.H. Rashmi, A. Raizda, G.M. Madhu, A.A. Kittur, R.Suresh & H.K.Sudhina). PVA assisted ZnO based mesoporous ternary metal oxides nanomaterials : synthesis,optimization, and evaluation of antibacterial activity (buzayeha abebe, H C Ananda murthy , Znyew zefera and Yeshaneh Adimas). Hexavalent chromium removal mechanism using conducting polymers(K.K. Krishnani, Sira Srinives , B.C. Mohapatra, V.M.Boddu,Jumin hoa , X.Meng , Ashok Mulchandani). Hexavalent chromium removal by using synthesis of polyaniline and polyvinyl alcohol (Majid Riahi Samani , Parisa Ebrahimbabaie and Hamed Vafaei Molamahmood) . Zinc ferrite –PVA nanocomposite and removal of chromium from aqueous solution (Sonal Agarwal , Nakshatra Bahadur Singh) .Degradation of micro and nanoplastics by photo catalytic methods (Bratovic A). Photodegradation and photostabilization of polymers,especially polystyrene (Emad Yousif and Raghad Haddad) . Influence of zinc oxide nanoparticles on structural and electrical properties polyvinyl alcohol films.(Ren , Y., Zhang, Y., Wang, H. Y. & Luo,C). An insight into the effect of Zinc oxide nanoparticles on the structural, thermal, mechanical properties and antimicrobial activity of Cs/PVA composite (A.M. Hezma , A.Rejab , Mohammed A. Manaa). Degradation of the PVA may also guide the sustainable development (Ren, Y., Zhang, Y., Wang, H. Y. & Luo,C.). Polyvinyl alcohol driven silver nanosheets as nanocatalyst for fast degradation of methyl orange dye (Pirzada; Ashfaq Hussian;Ali Shah;Syed Farman;Srijuddin; Sangah; Ali Asghar; Mugheri, Abdul Qayoom;Mahmoud,Mohamed H;Tashish,Waddah Mouhammad; Akhtar, M. Shaheer). Preparation of PVA based composites with the addition of Zinc Oxide nanoparticles (Jolant Pulit-Prociak; Krzysztof Pszczółka ;Jaroslav chwastowski; Paweł Staroń; Anita Staroń; Elżbieta Sikora ;Sławomir Michałowski ; Marcin Banach).

Recent advance in the biodegradability of PVA and its derivative material(Zhang, H. Z., Liu, L. & Luo, R.). Removal and adsorption characteristics of PVA from aqueous solution using electrocoagulation (Chou,W. L). Screening and identification of strains Degradation PVA (Guo, L. L., Li, S. N. & Wang, S .X.).

CHAPTER 3

AIM AND SCOPE

The aim of this project is to prepare polymer composite with metal and ZnO as nanocatalyst. Polymer (unstable polymer) is used as a catalyst for hexavalent chromium reduction. PVA is used to reduce Cr^{6+} . Polymer and particularly plastics are subjected to degradation at all stages of their product life cycle, including their production, use, disposal into the environment and recycling. The reduction of hexavalent chromium using destabilized polymer is carried out here. Further the composite with incorporated zinc oxide nanoparticles were analyzed and characterized by XRD and DLS analysis. The zinc oxide nanoparticles were blended with the prepared polymer gel and this solution was characterized by FTIR analysis.

CHAPTER 4

MATERIALS AND METHODS

- PVA (POLYVINYL ALCOHOL)
- DISTILLATED WATER
- CONCIAL FLASK
- MAGNETIC STIRRED
- SHAKER
- COPPER SULPHATE
- POSTASSIUM DICHROMATE
- BEAKER
- MAGENTIC PELLETS
- WATCH GLASS
- HEAT OVEN
- STANDARD FLASK
- PHOTOCOLORMETRIC MACHINE
- WIEGHING MACHINE
- ZINC OXIDE
- HCl (HYDROCHOLORIC ACID)ACIDIC CONDITION
- ZINC CHLORIDE
- SODIUM HYDROXIDE
- WATTMAN FILTER PAPER

4.1 PERPARATION OF PVA FILM

Taking up the PVA sample with the distilled water and leaving for stirring for 2 hours and its becomes jelly form, by that we are adding ZnO. It used as catalyst for reduction process. ZnO nanoparticles are prepared by mixing a solution of Zinc Chloride (1g) dissolved in 20 ml water to a solution of NaOH taken in 1:1 ratio. The precipitate formed is washed several times with water and dried in an oven for 12 hours and subjected it to a temperature of 240°C in a muffle furnace. 1g of the ZnO nanoparticles prepared is added to a solution of 2g of PVA (polyvinyl alcohol) dissolved in 50 ml water to prepare the PVA-ZnO composite.

4.2 PVA REDUCTION IN CHORMIUM HEXAVALENT

We were trying to degradate polymer material by incorporating nanocatalyst or nanomaterial. ZnO used as a nanocatalyst. By incorporating nanocatalyst, In IR spectrum we were able to see much difference, that would be possible for degradation, due to the polymer stability is becoming less. PVA is polymer, every polymer consider to be highly stable. We are incorporating ZnO by polymer.



Fig : 4.1 synthesis of catalyst in chromium

After the reduction polymer, We were testing its stability by incorporating ZnO to make unstable and decreasing its stability. This happened using hexavalent Chromium reduction by using incorporated ZnO + PVA. Stable polymer doesn't react in any type of reaction due to its high stability. We were testing its stability by chromium reduction, by reducing its state from Cr^{6+} to Cr^{3+} . So PVA + ZnO is reacting in the reduction process. Incorporated ZnO is making PVA (poly-vinyl alcohol) less stable by chromium reduction. The next day it was kept in a water bath for the supply of heat, for better synthesis of the ZnO nanoparticles. The solution was then centrifuged to get the pellets. The pellets obtained were then dissolved with distilled water and again centrifuged. The pellets hence obtained were the ZnO nanoparticles which were further used for other analysis and experiment.



Fig : 4.2 before the synthesis



Fig : 4.3 after the synthesis

This sample undergoes chromium reduction. Sample 1 is considered of PVA + ZnO in chromium solution, this reduction happens in 7 to 8 hours, so that PVA is losing its stability by ZnO as a nano-catalyst. PVA involves in a reduction reaction, when ZnO is present with PVA. PVA doesn't involve in chromium reduction. But I have incorporated ZnO to make it less stable and we have proved that chromium reduction is happening because of ZnO incorporation in PVA.

4.3 PREPARATION OF PVA (POLY VINYL ALCOHOL) METAL

Taking the sample PVA with 2g mixing the sample with 10 ml of distilled water and metal (CuSO_4) to make polymer metal . series of PVA-based compositions with zinc oxide, silver and copper nanoparticles were prepared. Mixing up this solution in magnetic stirrer for 24 hrs and filtrating the solution through wattmaan filter paper, the liquid solution comes outs and solid content will settle down in paper. Let the settled solution keep in heat oven for 24 hours and the solution is dried .



Fig : 4.4 preparation of pva + metal

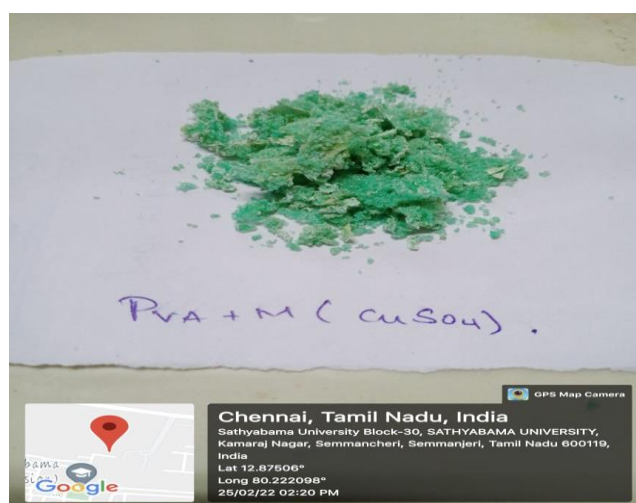


Fig : 4.5 pva (metal)

4.4 PREPARATION OF PVA (METAL) IN NANOCATALYST

The metal polymer is prepared by combining the mixture of copper sulphate and PVA. The solution of nanoparticles of ZnO in PVA(Metal) is prepared by mixing of Zinc oxide powder with distilled water with constant stirring over the magnetic stirrer for complete dissolution of the particles in the water without any formation of lumps. On the other hand, PVA was also added to distilled water and was again stirred over magnetic stirrer. These two solutions were mixed together and made into single solution. It was then kept over the magnetic stirrer for about 20 hours for the complete mixing of solution. And the solution filtered and kept in heat oven for drying. The synthesized zinc oxide nanoparticles dissolved and mixed with the hydro gel thoroughly.

4.5 CHARACTERIZATION OF A NANOPARTICLES

The prepared zinc oxide nanoparticles were characterized following methods

4.5.1. ULTRAVIOLET-VISIBLE DIFFUSE REFLECTANCE SPECTROSCOPY (UV-Vis DRS) ANALYSIS :

According to this technique, it uses ultraviolet (UV), visible, or infrared (IR) light as a probing medium. The interaction of light with strongly absorbing materials, it occurs in the first 10-20 nm. For this purpose, the feasibility of UV-Vis diffuse reflectance spectroscopy (UV-Vis DRS) is described as a rapid, inexpensive and non-destructive method of analysis of PM filters, UV-Vis spectrometers can measure both diffuse and spectral reflectance without using special attachment. The formation of a surface layer as a result of environmental interactions can thus be studied in situ. The measurements are quick: a complete differential reflectogram, that is, an automatic scan from the UV through the visible to the IR region, takes about 3 minutes. Thus the differential reflectometer probes 50-100 atomic layer into non transparent solid surfaces.

4.5.2 Fourier Transform Infrared Spectroscopy (FTIRs) ANALYSIS :

Fourier Transform Infrared Spectroscopy (FTIR) is a technology used to get an infrared spectrum of the absorption or emission of solid, liquid or a gas. An FTIR spectrometer concurrently collects the high-resolution spectral data over a wide range of spectra. This has a significant advantage over a dispersive spectrometer, which measure the identity over a narrow range of the wavelength at a time. FTIR analysis of zinc oxide nanoparticle sample was performed with technologies portable attenuated total reflectance (ATR) Fourier transform infrared (ATR-FTIR). Sample spectra were recorded in the middle infrared range from. This features out the increase in the crystalline phase of the PVA matrix after embedding of Zn particles. FTIR spectra of pure PVA and PVA-zinc nanoparticles films in the wave number range 600-1800 cm.

4.5.3 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS :

SEM is a surface imaging method in which the incident electron beam scans across the sample surface and interacts with the sample to generate backscattered and secondary electrons that used to create an image of the sample. The functional groups present in the films were identified by a thermo nicolet avatar 370 Fourier transform infrared (FTIR) spectrometer with a spectral range of 4000-400 cm $\text{\AA}$ 1 and resolution of 4 cm $\text{\AA}$ 1. The SEM images of ZnO and PVA metal.

4.5.4 THERMO GRAVIMETRIC ANALYSIS (TGA) ANALYSIS :

Thermo Gravimetric Analysis (TGA) was carried out using thermo gravimetric analyzer (TGA) in atmosphere (2960 SDT V2.2B). Sample was scanned from 10-850°C at a heating rate of 10°C min⁻¹. Changes in the sample weights were recorded electronically by change in voltage output from a linear variable differential transformer. The first derivative of the primary thermo gravimetric curves, representing rate of weight loss, were determined from tangents to the weight loss curves.

4.5.5.ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDAX) ANALYSIS

Energy dispersive X-ray spectroscopy, sometimes called Energy dispersive X-ray analysis , is an analytical technique used for the elementary analysis or chemical

characterization of a sample. It interaction of some source of X ray excitation and a sample. They demonstrated this EDAX pattern of synthesized zinc oxide nanoparticles at 25°C . The EDAX pattern of zinc oxide nanoparticles and they obtained a value of approximately KeV, which is a characteristic peak of zinc oxide nanoparticles .

CHAPTER 5

RESULTS AND DISCUSSION

5.1 NANOPARTICLES INCORPORATING BY METAL (COPPER SULPHATE)

Metallic nanoparticles (MNPs) have a metal core composed of inorganic metal or metal oxide that usually covered with a shell made up of organic or inorganic material or metal oxide. This is used in sunscreen, skin protection. Polymer is soluble in water. By incorporating it losses its stability. By adding of ZnO in PVA metal for chromium reduction for losses polymer stability by hexavalent chromium reduction. The formation of metal nanoparticles depended on the functional groups and charge on the surface of the polymer particles. The metal nanoparticles were formed due to the reduction of metal ions, accompanied by the reduction of MNPs.



Fig : 5.1 mixture of metal and nanoparticles.

5.2 Preparation of PVA(Metal) with Nanoparticles

The solvent was prepared with the combination of PVA(Poly Vinyl Alcohol) ,CuSo₄ (copper sulphate), ZnO(Zinc oxide) and Distilled water.

For 2 grams of PVA,
CuSo₄: 1 mg
ZnO : 0.75 mg
Distilled water: 25 ml

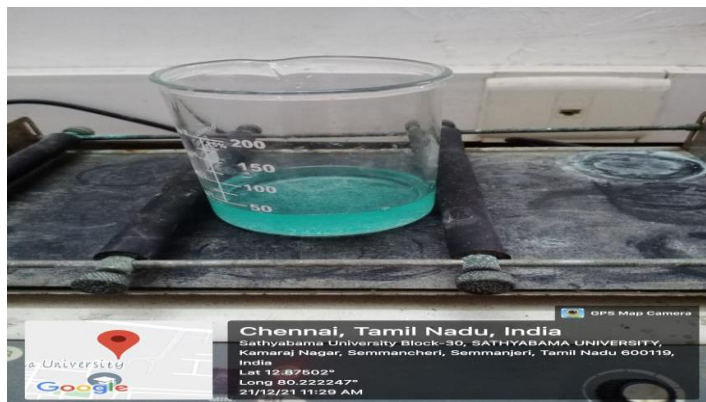


Fig : 5.2 (A) : pva+metal preparation



Fig : 5.2 (B) : pva(metal)+zno

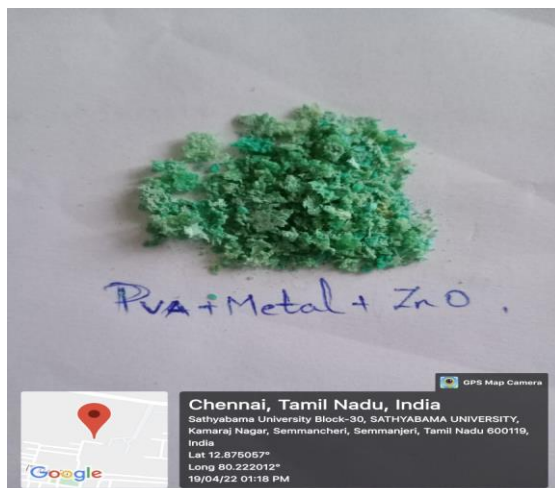


Fig : 5.2 (C): final sample of PVA(metal)+ZnO

5.3 SYNTHESIS OF CHROMIUM IN PVA METAL WITH ZnO CATALYST

5.3.1 PREPARATION OF PVA METAL

PVA : 2mg

Metal (CuSO_4) : 1mg

Distillated water : 25 ml

5.3.2 PREPARATION OF CHROMIUM SOLUTION

CHORMIUM : 1mg

Distillated water : 100mg

Filling the chromium in the 100 ml standard flask and making upto 100 ml, from that we are taking 0.4N, 0.6N, 0.8N, 0.12.

As a source of hexavalent chromium, potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) is employed. Dissolve 2.8287g potassium dichromate in 1000ml distilled water to make a Cr(VI) stock solution with a concentration of 1000 mg/l. The solution is diluted to the desired concentration. Cr solutions with a concentration of 10-15 mg/l (VI). The pH of the water

was altered. Sodium hydroxide and sulphuric acid were used. For synthesis the PH and temperature conditions.

We were testing its stability by incorporating ZnO to make unstable and decreasing its stability. This happening using hexavalent Chromium reduction by using incorporated ZnO + PVA . Stable polymer don't reacts in any type reaction due to highly stable and stability. We were testing its stability by chromium reduction , by reducing it state from Cr⁶⁺ to Cr³⁺. So PVA+ ZnO is reacting in reduction process. Incorporated ZnO is making PVA (poly-vinyl alcohol) less stable by chromium reduction.

According to previous studies, the consummation of Cr(VI), took place into two successive steps: a fast step, the first 30 minutes, 196 Proc. corresponding to the reduction of chromium (VI) into chromium (V) by a one electron charge transfer from the polymer matrix to Cr(VI) in its excited state and slow step corresponding to the reduction of chromium (VI) into chromium.

5.4 ANALYSIS OF HEXAVALENT CHROMIUM

The concentration of hexavalent chromium ions in the sample is evaluated spectrophotometrically by producing a red-violet colour in an acidic condition solution with 1,5 di phenyl carbazide as a complexing agent. After 20 minutes, the absorbance of the red-violet solution is measured at 530 nm-540 nm.



Fig : 5.3 photocalorimeter

5.5 VARIOUS CONDITIONS OF DEGRADATION

I have checked its Solubility with different conditions. By PH and temperature conditions,

5.5.1 PH CONDITIONS(ACIDIC /BASIC / NETURAL

5.5.1.1 ACIDIC CONDITION

I have taken small amount of sample of PMZ , we able to see some changes in this conditions . Metals is getting dissolved. After few hours it becomes colourless HCl is used in this condition. See the given Figure below.

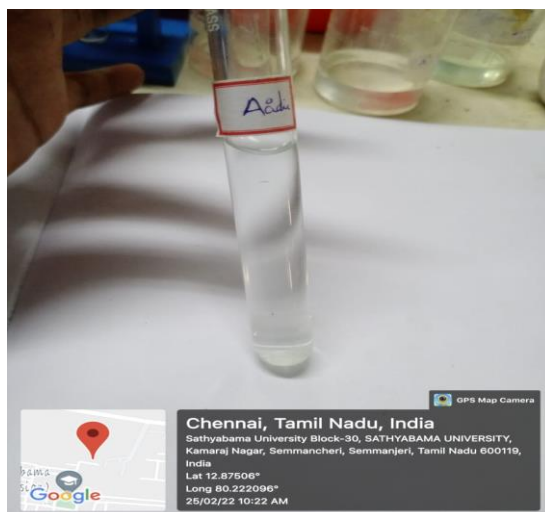


Fig : 5.4 Acidic condition reduction

5.5.1.2 BASIC CONDITION

I able to have changes differ to acidic conditions, sample gets settled in the solution, and after few hours it become darker green. NaOH is used for this condition. See the Figure given below.



Fig : 5.5 basic condition reduction

5.5.1.3 NETRUAL CONDITION

The sample does not dissolved and remains same and no colour changes happen. See the Figure given below.

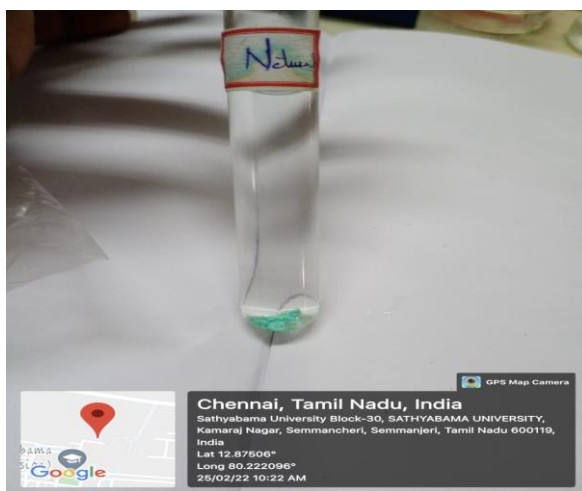


Fig : 5.6 netural condition reduction

From these conditions, we concluded, in natural condition its remains same and more stable .



Fig : 5.7: pH conditions

5.5.2 TEMPERATURE CONDITIONS

5.5.2.1 AT 40°C (ACIDIC /NETRUAL/ BASIC)

Acidic (HCl): Becomes colourless and metals get dissolved within few minutes

Basic (NaOH): Becomes darker green and metals get settled and colour changes faster within few minutes .

Neutral : Its remains same, no colour changes and metal with polymer gets stable.

From these conditions , neutral remains same.



Fig : 5.8 acidic condition at 40 °C



Fig : 5.9 neutral condition at 40 °C



Fig : 5.10 basic condition at 40 °C

5.5.2.2 AT 60° C (ACIDIC /BASIC /NETURAL)

Acidic (HCl): becomes colourless and metals gets dissolved within few mintues at high temperature

Basic (NaOH): becomes darker green and metals gets settled within few mintues at high temperature .

Netural : remains same . No changes happened and get stability than basic

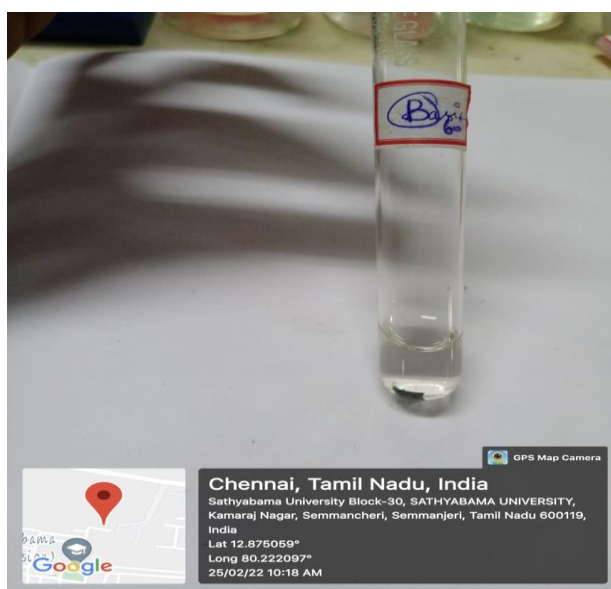


Fig : 5.11 basic condition at 60 °C



Fig : 5.12 acidic condition at 60 °C



Fig : 5.13 neutral condition at 60 °C

5.6 CHROMIUM REDUCTION OF NANOPARTICLES

Composite as a catalyst, Chromium reduction. Adding polymer in chromium VI to lose its stability with different weight of sample and different soluble of Cr solutions. By this process, polymer loses its stability and dissolved in the solution. By 0.03 and 0.04g weighing of PMZ is differs by time and colour of the sample. 0.03g weight sample, reduction happens faster than 0.04 g, with 10 ml of solution. This is due to chromium reduction is happening in the PMZ sample by degradation and adsorption.

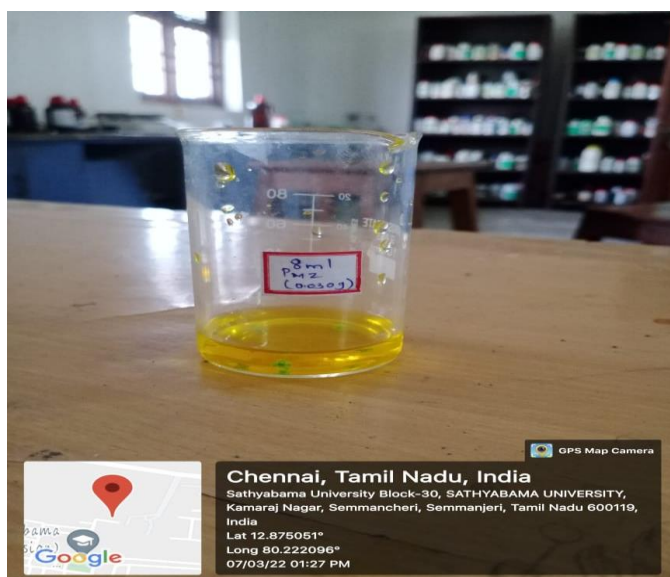


Fig : 5.14 8ml of chromium reduction

Table 5.1 EFFECT OF CHROMIUM REMOVAL FOR THE INITIAL CONCENTRATION OF 0.0326 N

PARAMETERS (8ml)	OD VALUE FOR THE CATALYST DOSAGE OF 0.03g	OD VALUE FOR THE CATALYST DOSAGE OF 0.05g
INITIAL	0.28	0.33
AFTER 1Hr	0.23	0.31
AFTER 1Hr	0.17	0.25
AFTER 1Hr	0.15	0.22
FINAL(after a day)	0.03	0.07
Removal(initial – final/initial*100)	89.28	78.78

Table 5.2 EFFECT OF CHROMIUM REMOVAL FOR THE INITIAL CONCENTRATION OF 0.0489 N

PARAMETERS(12ml)	OD VALUE FOR THE CATALYST DOSAGE OF 0.03g	OD VALUE FOR THE CATALYST DOSAGE OF 0.05g
INITIAL	0.39	0.39
AFTER 1hr	0.35	0.33
AFTER 1hr	0.31	0.29
AFTER 1hr	0.40	0.25
Final (after a day)	0.40	0.11
Removal (initial – final/initial*100)	NO CONSIDERABLE OD CHANGE WAS OBSERVED.	71.79



Fig : 5.15 12ml of chromium reduction

5.7 CHARACTERIZATION OF PMZ CATALYST

5.7.1 ULTRAVIOLET-VISIBLE DIFFUSE REFLECTANCE SPECTROSCOPY (UV-Vis DRS)

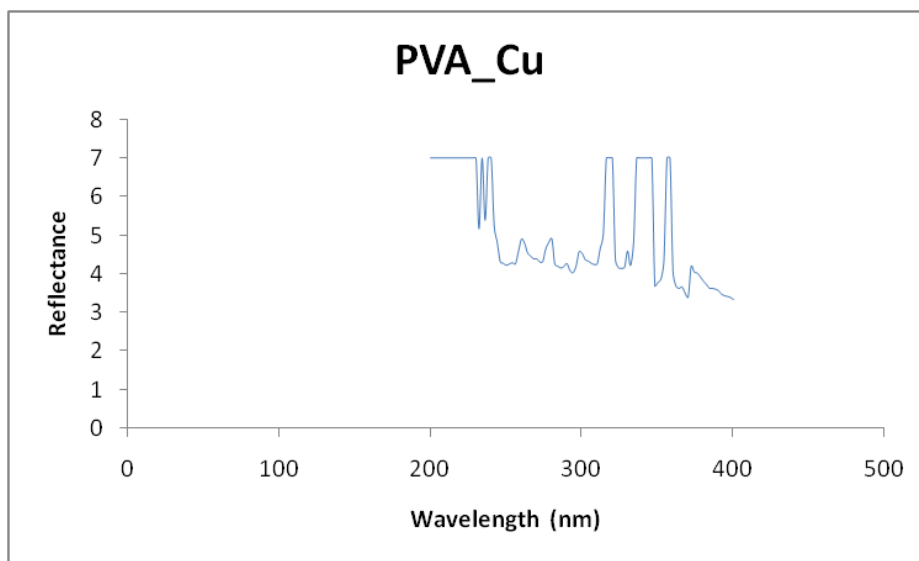


Fig: 5.16 PVA (METAL) UVDRS

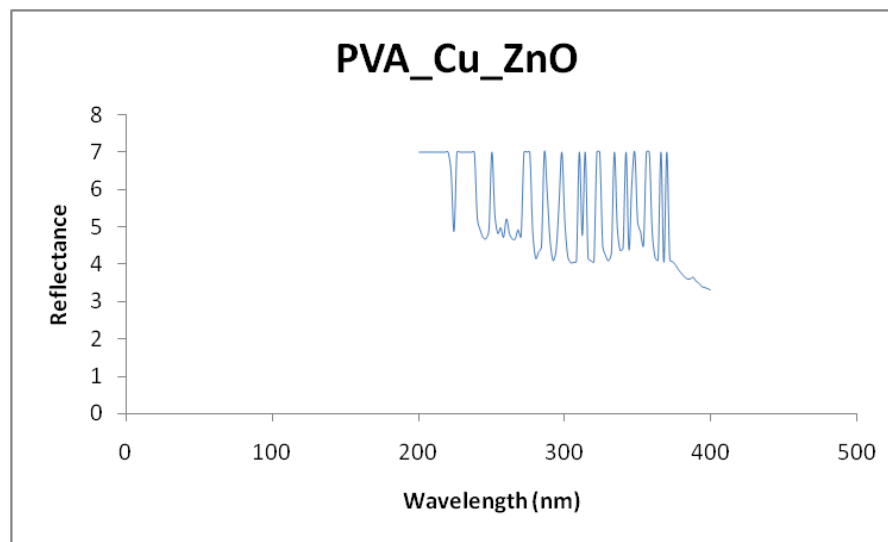


Fig: 5.17 PVA (METAL WITH ZnO) UVDRS

In order to obtain more information about the nanocomposite synthesized, UV-DRS spectra for the PVA-Cu and PVA-Cu-ZnO composite were recorded. The UV-DRS spectra of PVA-Cu-ZnO shows more spectral lines in the in the range of 200 -400 nm as compared to PVA-Cu. The calculation of the band gap energy from the Tauc Plot is scope of the future study.

5.7.2 Fourier Transform Infrared Spectroscopy (FTIRs) ANALYSIS

The FTIR analysis was used to determine the possible functional groups present in the synthesized PMZ nanoparticles. . FTIR spectra show major peaks at 3853, 2090, 433, 422, 416 and 410 cm^{-1} . They were compared to the bond angles extending C-S linkages-H stretching-O-C group-H bond, alkyl ketone, alkane group, aromatics group. FTIR spectra of PMZ nanoparticles. The main IR band of pure PMZ was reported in Figure.

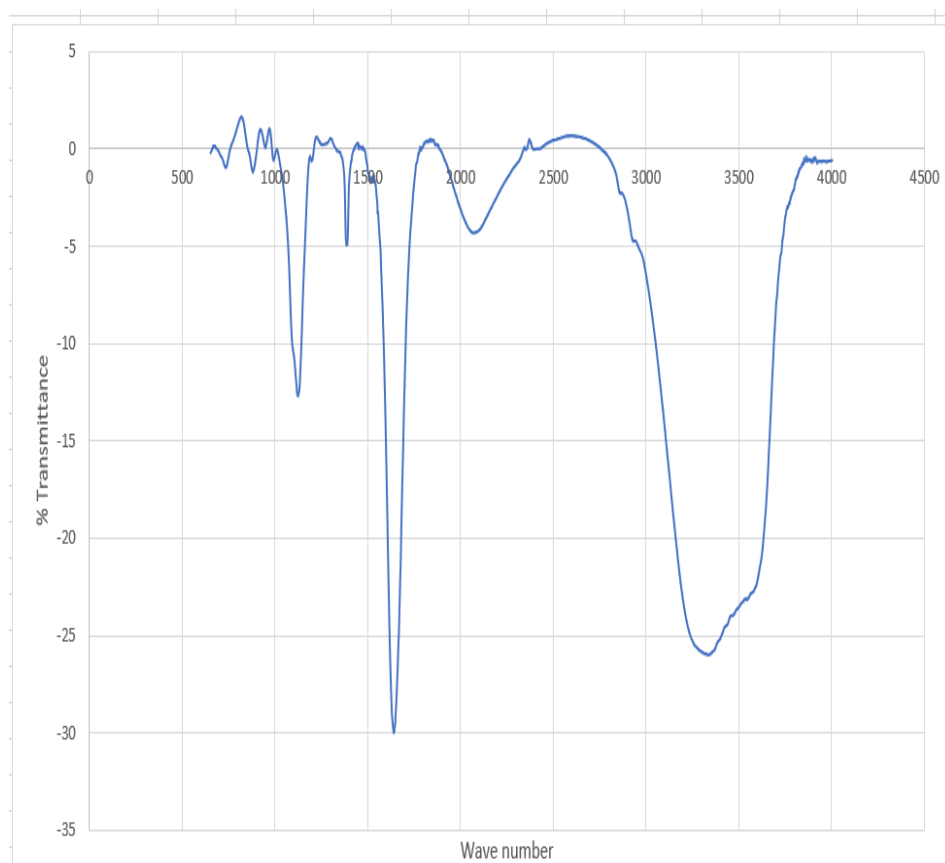


Fig : 5.18 FTIR of PVA (METAL) WITH ZnO IMAGES

5.7.3 THERMO GRAVIMETRIC ANALYSIS (TGA) ANALYSIS:

Thermodynamic stability of a material can be assessed using TGA. If a species is thermally stable within a given temperature range, no mass change can be seen. Little or no slope in the TGA trace indicates negligible mass loss. The TGA method also determines a material's maximum use temperature. The material will begin to disintegrate once it reaches this temperature. TGA is a polymer analysis technique. Because polymers typically melt before decomposing, TGA is primarily used to investigate polymer thermal stability. Before 200 °C, most polymers melt or degrade. TGA can analyse a class of thermally stable polymers that can withstand temperatures of at least 300 °C in air and 500 °C in inert gases without structural changes or strength loss.

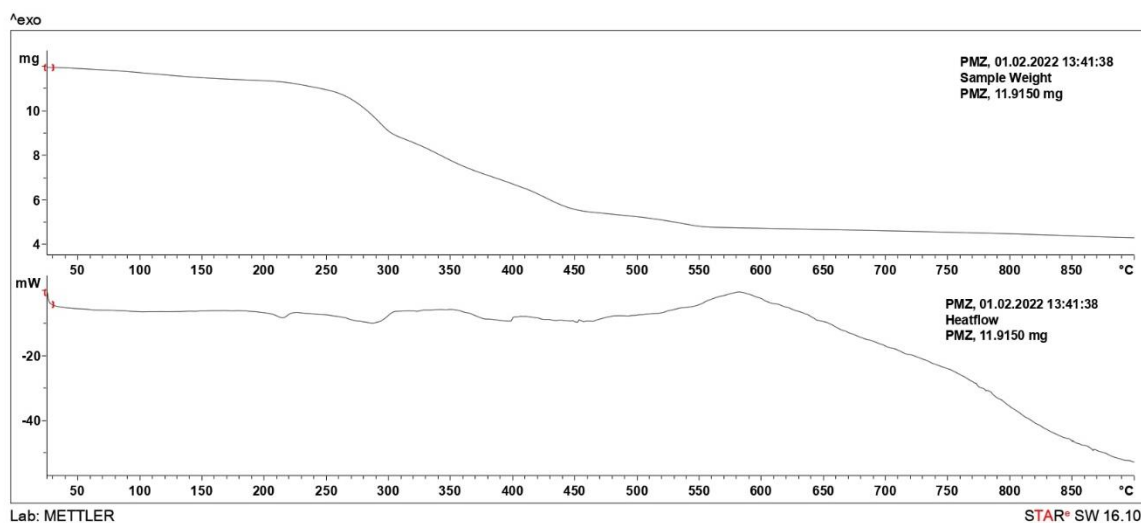


Fig : 5.19 TGA of PVA (METAL) WITH ZnO IMAGES

5.7.4 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS:

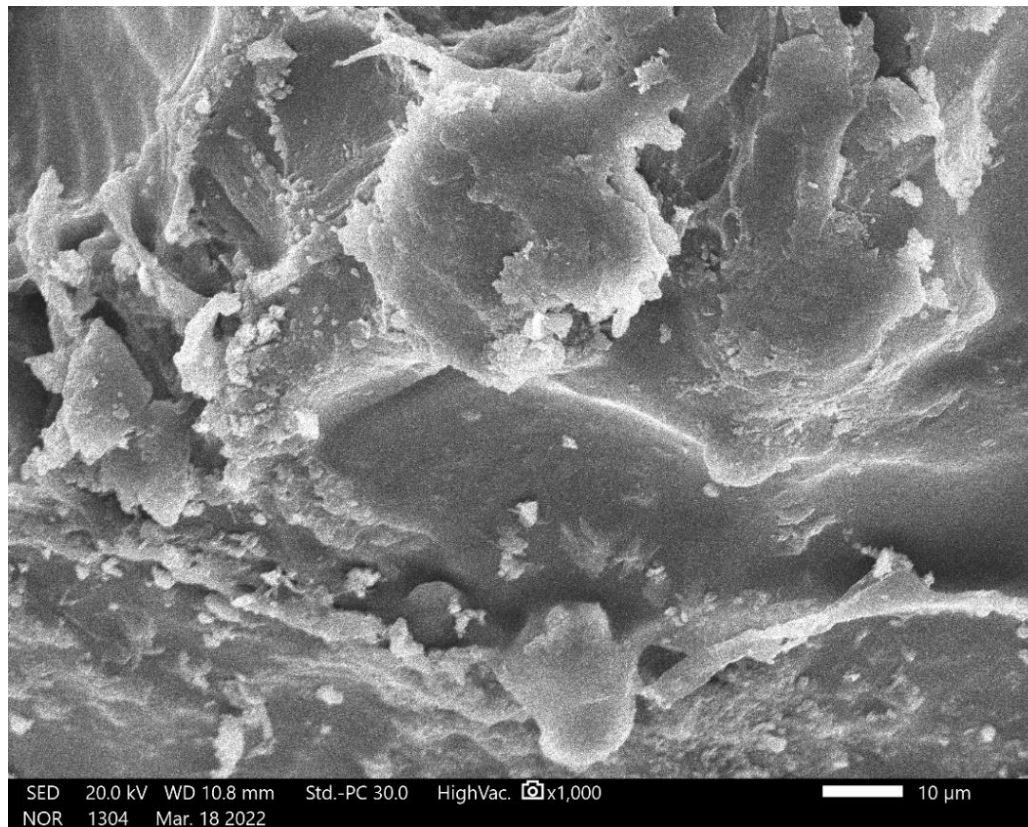
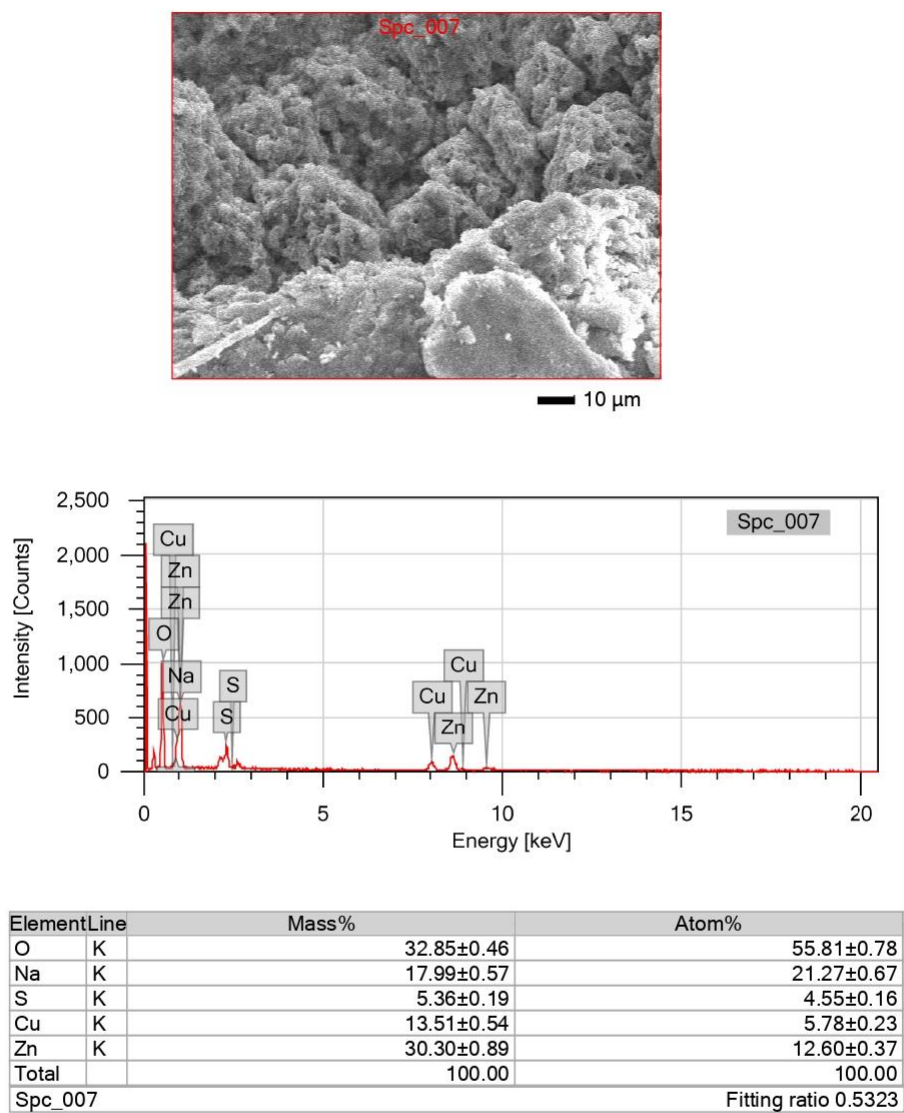


Fig : 5.20 SEM of PVA (METAL) WITH ZnO IMAGE

5.7.5 ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDAX) ANALYSIS:



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Fig : 5.21 EDAX of PVA (METAL) WITH ZnO IMAGES

SUMMARY AND CONCLUSION

The persistent nature of polymer and their infinitesimal size with potential lethal consequences is being noticed by researchers and societies across the world and new means of degradation of these plastics to save the earth's environment from getting further polluted is the need of the hour. Thus, in this work, a major focus was on the analysis and development of the scientific principles and experimental techniques to accelerate the break-down of documented polymer reduction pollutants particularly PVA through heterogeneous nanocatalysis process using high surface area designer nanocatalysts, namely Zinc oxide (ZnO) and METAL (CuSO₄) coated Zinc oxide). The optical properties of ZnO were modified using metal (Zn-NPs) to improve the overall plasmonic nano catalyst performances by lowering down the recombination processes and improving the visible light absorbance for application of visible light nanocatalysts using sunlight. In summary, slight hydrogenation retained the tubular structures with triphase composition in ZnO. The above results show that the slightly hydrogenated triphase ZnO nanotubes can, without the need of a catalyst, be intrinsically activated to show a high Cu₂ evolution rate in nanocatalytic degeneration. 50 µm commercial PVA film was irradiated for 175 hours with visible light in the presence of nanocatalysts (ZnO and Zn-ZnO). Changes in the properties of PVA were observed through FTIR spectroscopy, DMA, and Optical microscope and SEM. Formation of low molecular weights of various functional groups after nanocatalytic degradation was observed; characteristic peaks within the range of 3600-3610 cm⁻¹, 1700-1760 cm⁻¹, 1100-1300cm⁻¹, and 880-920 cm⁻¹ revealed the formation of hydro-peroxides, peroxides, and unsaturated groups respectively in the FTIR spectra of PVA film. The presence of higher CI indicated the higher degree of oxidation and in directly confirming the disintegration of PVA upon visible light exposure. SEM results showed that the mechanical damages in the polymer matrix occurred following nanocatalytic degradation process.

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