



SYNTHESIS AND CHARACTERIZATION OF COPPER-CERIUM METAL NANO-COMPOSITES AND THEIR METAL GLYPTAL POLYMER

Mandal P. V.¹, Kathale N. E.²

1. P.G. Department of Chemistry, Sardar Patel College, Chandrapur, India
Email: pranav.mandal310@gmail.com, 9405125531

2. Department of Chemistry, Sardar Patel College, Chandrapur, India
Email: kathale.niren@gmail.com

Abstract

Copper-Cerium Metal Nano-composites and their units were synthesized in moderate dehydrated condition via conc. nitric acid in heated. Metal Glyptic Polymer of these Nano-composites was obtained by condensation of phthalic-anhydrides and glycerol in hydrogen peroxide environment. Metal-NCs reacts with glyptic to yields polymeric $\text{Cu}(\text{OEt})_2 \cdot \text{Ce}(\text{iOPr})_2$ Glyptal. Glyptal multi-dimensional structures having charge-balancing positive ions are hydrated in the cluster framework cavities. A statistical study of the effect on the polymerization process, the molar ratio of the component oxides and the water content of the mixture reveals that both the metal clusters specifically follows the studied parameters. The polymerization mechanism and structures of the products were investigated using conductivity, TGA, DCA analysis, XRD and SEM spectroscopy.

Keywords: Co-polymer, Copper-Cerium Metal Nano-Composites, $\text{Cu}(\text{OEt})_2 \cdot \text{Ce}(\text{iOPr})_2$ Glyptal, Thermostable, Hydrated fluxional cavities

I. Introduction

The strategy for incorporating metal-nanocomposites into polymers and macromolecular systems has often led to new properties and capabilities that would otherwise be challenging for purely-organic analogues to achieve. The increasing research activity in metal-nanocomposites and organometallic polymers is a testament to the opportunity for advancing our fundamental understanding of

polymer science and for triggering innovation in various technologies (1,2). This trend can be linked to $\text{Cu}(\text{OEt})_2 \cdot \text{Ce}(\text{iOPr})_2$ Glyptal polymer, the toughening of the legislation in terms of fire hazards combined with the growing use of flame retardants. The flammability behavior of polymers is defined on the basis of several processes and parameters, such as burning rates (solid degradation rate and heat release rate), spread rates (flame, pyrolysis, burn-out, smolder), ignition characteristics (delay time, ignition temperature, critical heat flux for ignition), product distribution (in particular, toxic species emissions), smoke production, etc (7,8). Our goal is then to inhibit or even suppress the combustion process acting chemically and physically in the solid, liquid or gas phase. We can interfere with combustion during a particular stage of this process, e.g. during heating, decomposition, ignition or flame spread. Three approaches can then be considered to reduce the flammability of polymers (9-11).

In organic synthesis, fire retardants and conducting material industry play a vital role in a growth of our nation; thus, its improvement can help economic growth by increasing the efficiency of organic reagents compounds, polymeric conducting materials and other product. In this research article, nanocomposites metal glyptal polymers, a new class of polymers, having a fundamental role in improvement of polymer, because they have better thermal and conductivity properties and they also act as better organic reagents than the pure glyptal polymer (6).

II. Materials and Methods

2.1 Materials

Copper Chloride-(CuCl₂·2H₂O) M. wt.170.48, Qualichem Laboratory (India), Cerium Sulphate-[Ce(SO₄)₂·4H₂O], Burogyne Burbidge's & CO (India) Mumbai, Hydrogen peroxide- (H₂O₂) M. wt. 34.01 g/mol, Isobutyl alcohol- (C₄H₁₀O) M. wt.74.122 g/mol, Phthalic-anhydride -C₈H₄O₃ M. wt.142.12g/mol, Toluene- (C₇H₈) M. wt.92.14 g/mol, Glycerol - C₃H₈O₃ M. wt.92.09 g/mol was procured from Thomas Baker (chemicals) pvt. Ltd. Mumbai). All chemical and reagents has research grade.

2.2 Synthesis of Nanoparticles

In this research, synthesis of organo-metallic nanoparticles is carried out via the oxidative addition reaction on central metal atom by approaching the organic monomers in inert condition. In this synthesis, the vacant d orbital's configurations of Cu-Ce metal atom are used and the synthesis of Cu(OEt)₂.Ce(iOPr)₂ is done in excess amount of ethanol and iso-propanol. The mixture was refluxed for 5 hours in round bottom flask with water condenser. After refluxing, the mixture was decanted in china dish and dried in hot oven at 80° C.



Fig:-01 Nano-composites of Ce(iOPr)₂



Fig:-02 Clusters of Cu(OEt)₂

2.3 Synthesis of Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer

In these syntheses, nanoparticles were fused in glyptal-polymer by condensation polymerization method. Very small amount of Cu(OEt)₂.Ce(iOPr)₂ nano-composites (0.0250mg+ 0.0249mg) are induced in 3.5 g/mole glycerol treated with 8.5 g/mole phthalic

anhydride of the mixture was first heated vigorously on direct flame in china dish (6). After well mixing Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer become which is viscous, then dried over the sand bath at 125oC to 135oC. Depending on the ratio of nanoparticles various proportions of Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer are obtained.



Fig:-03 Cu(OEt)₂.Ce(iOPr)₂ Glyptal (Low)

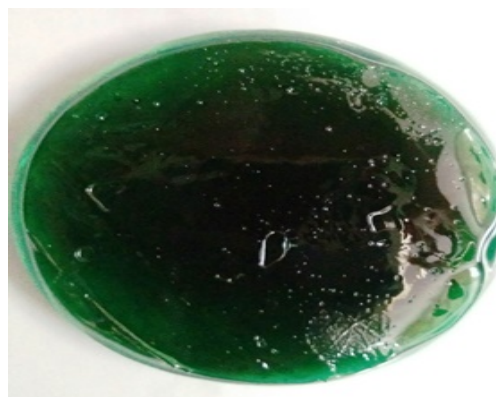


Fig:-04 Cu(OEt)₂.Ce(iOPr)₂ Glyptal (High)

III. Characterization and Data Analysis

3.1 XRD Analysis Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer

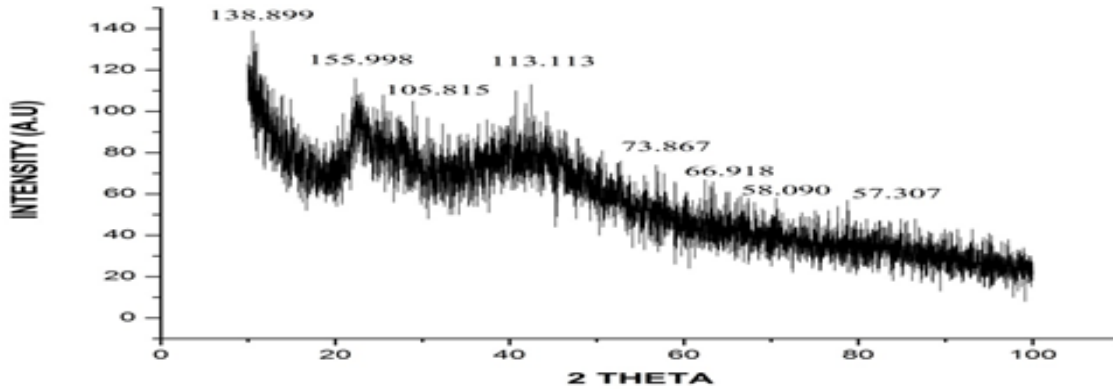
3.1.1 Calculation of Particle Size from XRD Data

From the XRD data, considering the peak at degrees, average particle size has been estimated by using *Debye-Scherer formula* (3)

is $D = 0.9 \lambda / \beta \cos\theta$. Inter-planar spacing between atoms (d-spacing) is calculated using *Bragg's Law* (4) is $2d \sin \theta = n \lambda$ and enumerated in Table 02.

Where, λ is wave length of X-Ray (0.1540 nm), β is FWHM (full width at half maximum), θ is diffraction angle, d is d-spacing and D is particle diameter size.

XRD analysis, Main Graphics view: Nano-composite (Cu.Ce) :



Graph:- 01 Cu(OEt)₂.Ce(iOPr)₂Glyptal polymer

Pos. [°2Th.] (θ)	2 θ	Cosθ	sin θ	FWHM [°2Th.](β)	βcosθ	Size of particles (D)(nm)	Rel. Int. [%]
5.059	10.1185	0.3397	0.9405	0.152	0.0516	2.6860	89
11.7618	23.5237	0.6934	0.7205	0.285	0.1976	0.7014	100
21.7517	43.5034	0.9714	0.2371	0.241	0.2341	0.5920	72.50
29.4809	58.9619	0.3562	0.9343	0.277	0.0986	1.4056	47.35
39.8263	79.6527	0.5281	0.8491	0.321	0.1695	0.8176	36.73

Table:-02

3.2. SEM Analysis of Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer

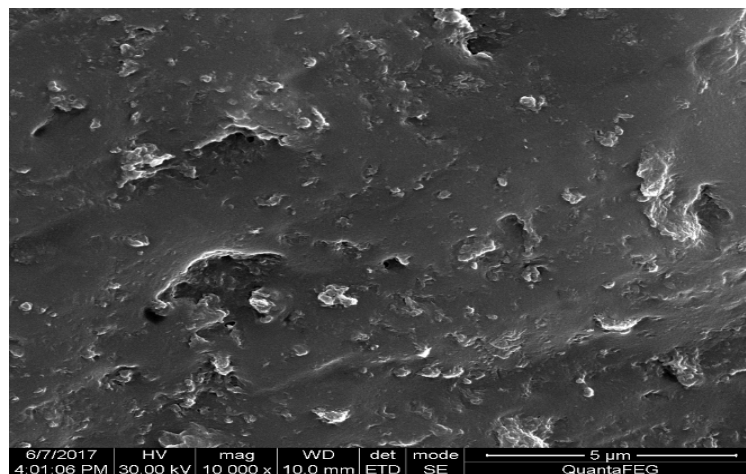
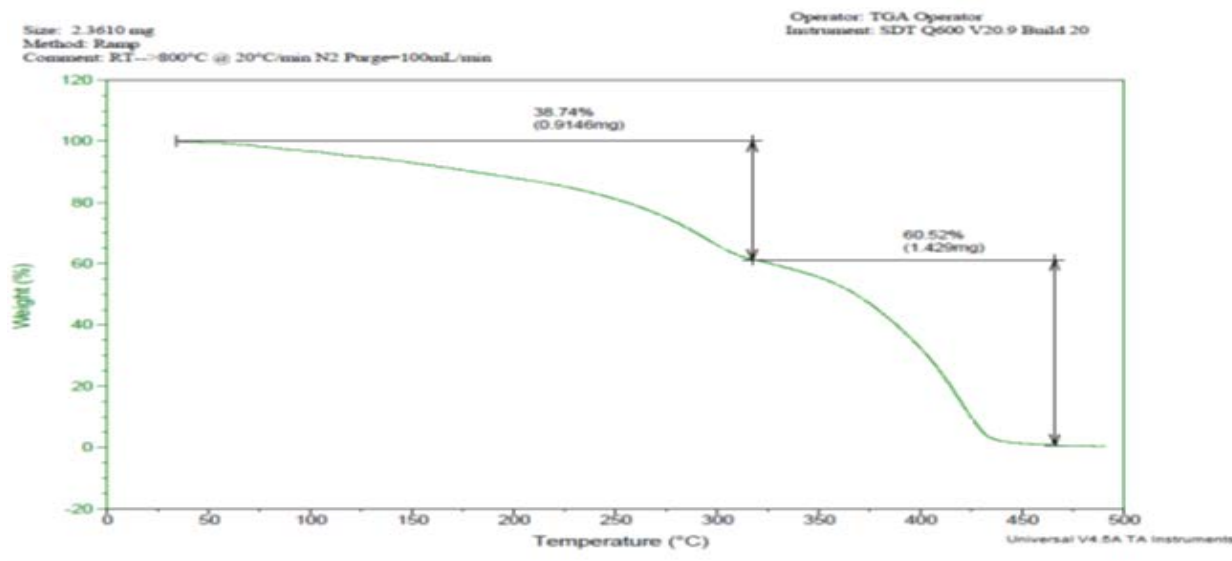


Fig.05

3.3. TGA Analysis of Cu(OEt)₂.Ce(iOPr)₂ Glyptalpolymer



Graph:- 02

IV. Results and Discussions

4.1 SEM, TGA and Analysis of Cu(OEt)₂.Ce(iOPr)₂ Glyptalpolymer

The miscibility between the organic polymer i.e. Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer phase and the nanocomposites phase was investigated by SEM and TGA. The SEM images of the polymer hybrids are in Fig. 05 and 06, show the phase separation at a magnification of about 10,000X. The white particles, whose size could be estimated to be around 0.7014nm, indicate a nanocomposites phase (7,8). On the other hand, in the case of the transparent hybrid polymers, the results suggest a homogeneous dispersion between the organic polymer and the nanocomposites phase about 200°C. The small apparent mass change at the end of the experiment is a corresponding to the furnace switching off. The thermal behavior of all the compositions was investigated by sequentially firing both powder and fragment samples at temperatures between 500–800°C for 20°C/min and determining the crystalline products formed at each temperature by XRD.

In TGA analysis graph 02, the weight of the sample is continuously recorded as a function of temperature. The hybrid organic polymer has change in weight of sample due to dehydration and decomposition as a function of temperature. In 315°C first decrease 60.52% of sample and maximum weight decreases of shown at 472°C i.e. 98.69% . it's results shows that the maximum stability in course of reaction

time as a reagents.

Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer almost insoluble in all organic solvent i.e. n-Hexane, Benzene, Toluene and water etc. but partially soluble in dioxane at temperature range 100-110°C. The very small amounts of metal nanoparticles when added to the metal glyptal polymer has been shown the partially conductivity. We have determined the melting point by open capillary method.

V. Conclusion

The XRD, SEM and TGA analysis ensures the insertion of metal nanoparticles in glyptal polymer. We have successfully synthesized Cu(OEt)₂.Ce(iOPr)₂ Glyptalpolymer in a versatile and bio safe approach, at room temperature. A single source catalyst, simple economic and environmentally safe which will make it suitable for various applications in organometallic reagent. Dispersion of nanoparticles between organic polymers improves the thermal stability, organometallic properties and surface phenomenon. XRD analysis have confirmed that synthesized particle are multiphase anatase Cu(OEt)₂.Ce(iOPr)₂ Glyptal polymer and their average nano size is approximately 0.7014 nm to 2.6860 nm. We expect that this synthesis technique would be extended to prepare many other important metal oxide nano structures.

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