

# An Experimental Study on the Thermal Properties and Electrical Properties of Polylactide Doped with Nano Aluminium Oxide and Nano Cupric Oxide

Abdul Mujeeb<sup>1</sup> · Anupam Glorious Lobo<sup>2,3</sup>  · A. J. Antony<sup>1</sup> · M. K. Ramis<sup>2</sup>

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**Abstract** The research on polylactide has been trending over the past 10 years as it is a versatile biopolymer which finds applications in food packaging and medical industries. In this research, nano aluminium oxide and nano cupric oxide have been impregnated into polylactide matrix using sonication and the nano composite thin films were prepared using solution casting technique. The thermal characterization of the neat and doped polymer films were conducted using thermogravimetric analysis and differential scanning calorimetry. The addition of nano aluminium oxide and nano cupric oxide increased the glass transition temperature and melting temperature of the samples in comparison with neat polylactide. Degree of crystallinity of all the doped samples increased with respect to the neat sample except for the sample doped with 1 mg nano aluminium oxide. The I–V characterization of the neat and doped samples revealed that addition of the nano powders reduced the resistivity of PLA by 35–45%. These results recommend the use of polylactide doped with nano aluminium oxide and nano cupric oxide as a potential semi-conducting polymeric materials.

**Keywords** Differential scanning calorimetry · Nano aluminium oxide · Nano cupric oxide · Polylactide · Thermogravimetric analysis · Resistivity

## Introduction

Polylactide, in comparison with other plastics produced from petroleum products, has been catching the eye of researchers all over the world mainly because of its biodegradability and processability from renewable resources. As a result, attempts are being made to improve the biodegradability (Tokiwa et al. 2009) and biocompatibility of polylactide to improve its sustainability and environmental safety (Kurtycz et al. 2013). Due to the poor mechanical and barrier properties exhibited by polylactide (Othman 2014), research has been dedicated to improve its mechanical and barrier properties by the addition of biocompatible nano particles like montmorillonite (MMT) (Ray et al. 2003), silver (Shameli et al. 2010), zinc oxide (Doumbia et al. 2015), titanium di oxide (Farhoodi et al. 2012; Ali and Noori 2014). Nano graphene oxide has proved to be highly compatible with polylactide/starch composites as it induced crystallization and improved mechanical and barrier properties at extremely low doping concentrations (Duo et al. 2016).

A research on PLA doped with talc and poly ethylene glycol revealed that the crystallinity of the composite significantly increases up to 43.3% due to the nucleating effect induced by 0.7  $\mu\text{m}$  talc particles in the presence of PEG (Tábi et al. 2014). The addition of multiamide nucleating agent has also found to favour PLA crystallization by increasing the overall crystallization rate and hence transforming PLA into a promising engineering material (Wei et al. 2012). However, no research work

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Abdul Mujeeb and Anupam Glorious Lobo have contributed equally to this work.

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✉ Anupam Glorious Lobo  
anupamlobo5@gmail.com

<sup>1</sup> Department of Mechanical Engineering, Sahyadri College of Engineering & Management, Adyar, Mangaluru, Karnataka, India

<sup>2</sup> Department of Mechanical Engineering, P.A. College of Engineering, Nadupadav, Mangaluru, Karnataka, India

<sup>3</sup> Door No 1-126A, Near Vishwamangala PU College, Pulinchady II Cross, Konaje, Mangaluru, Karnataka 574199, India

exploiting the thermal and electrical properties of polylactide doped with nano aluminium oxide and nano cupric oxide has been cited. The semiconducting properties of polylactide has not yet been extensively researched upon.

Thermal degradation of PLA in nitrogen atmosphere was conducted in order to propose kinetic models for isothermal and dynamic heating processes and quantified the heat sensitivity of PLA based on activation energy (Nicolae et al. 2008). Another study on the isothermal and dynamic thermal degradation regimes concluded that the activation energy of PLA samples increased with increasing molecular weight (Yang and Lin 2009).

The thermal stability of polylactide is also found to improve by the addition of particular nano particles or specific measure of the nano particles or both. Studies have been conducted on the thermal stability of polylactide by impregnating the same nano particle (sepiolite) in its modified and unmodified forms. Better results were obtained for unmodified sepiolite in contrast to the organically modified sepiolite (Liu et al. 2012). A DSC study on PLA-fumed silica (FS)-clay nano composite revealed that addition of 2 wt% clay to PLA FS composite increased the thermal stability and reduced the moisture weight percentage (Lai et al. 2015).

In order to meet the material design requirements, the glass transition temperature and crystallinity of polymers can also be lowered by the addition of plasticizers and nucleation agents which leads to the formation of small and imperfect crystallites (El-Hadi et al. 2002). The thermal degradation of polylactide in the presence of oxygen was studied between 70 and 150 °C where a reduction of molar mass resulting in variation in glass transition temperature, degree of crystallinity was observed. This work also derived a relationship between molar mass and strain at break and concluded that chain scission process is responsible for decrease of molecular weight of PLA (Rasselet et al. 2014). A study to determine the crystal structure of moulding grade polylactide revealed that polylactide achieved crystallinity up to 40% when annealed at 100–140 °C for 10 min (Tábi et al. 2010). The isothermal crystallization rate of PLA can be greatly enhanced by the addition of nano clay particles within the polylactide matrix. This work also studied the temperature effect on the crystallinity of the samples (Day et al. 2006).

Blending epoxidized palm oil (EPO) into the polylactide matrix has found to improve the degradation temperature, thermal stability as well as the mechanical properties of polylactide coupled with a considerable decrease in the glass transition temperature (Silverajah et al. 2012). Although metallic particles like Boehmite Alumina are expected to improve the thermal properties of polymers, the thermal stability of polylactide matrix showed no improvement when impregnated with

boehmite alumina particles in spite of the particles being well dispersed at lower loadings of boehmite alumina (Das et al. 2013).

The thermal characteristics of hot drawn PLA filaments have been investigated using modulated differential scanning calorimetry (MDSC) where it was found that modulated differential scanning calorimetry is a very powerful tool to analyse the thermal events of highly oriented polymer (Solarski et al. 2005). Apart from this, TGA techniques are useful in determining the degradation and volatilization patterns of biomass like pyrolysis oil obtained from areca tree (Bardalai and Mahanta 2016). DSC is also being used to determine the miscibility of polymers using different or same blending techniques (Cao et al. 2003). The results obtained from DSC also help in addressing manufacturing process related problems like destabilization of the polymer during injection moulding process (Tábi et al. 2010).

In this study, electrical current conducting nano particles like nano aluminium oxide and nano cupric oxide were doped into the polylactide matrix in 1 mg and 3 mg doping concentrations using ultra sonication and solution casting method (Lobo et al. 2016; Liu et al. 2012). The neat and the doped samples were analysed using thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and I–V characterization to study their thermal and electrical properties in order to exploit the applicability of the samples as semiconducting polymeric materials.

## Materials

Polylactide was purchased from Shanghai Guangue Bio-Tech Pvt. Ltd. Shanghai, China. The manufacturer claims the density of the material to be 1.27 g/cm<sup>3</sup>, melt flow index as 10–30 g/10 min and distortion temperature to be 70 °C. Nano aluminium oxide and nano cupric oxide was obtained from Sigma Aldrich. The nanoparticle size is less than 50 nm as specified by the manufacturer.

## Nanocomposite Preparation

### *Preparation of Neat Films*

0.375 g PLA was dispersed in 15 ml chloroform by sonicating the mixture for about 25 min. This mixture was poured into a petridish and set aside for a day for film formation. The formed film was the peeled and characterized.

### *Preparation of Doped Films*

PLA was dispersed in chloroform as mentioned above. To this solution, the nano powders (nano aluminium

oxide/nano cupric oxide) were added in their respective concentrations (1/3 mg). The solution containing nano aluminium oxide was sonicated for about 45 min and the solution containing nano cupric oxide was sonicated for about 55 min to ensure proper dispersion. The respective solutions were cast into the petridishes and set aside for film formation. The formed film was the peeled and characterized (Lobo et al. 2016).

**Thermogravimetric Analysis (TGA)**

TGA analysis was carried out to study the thermal degradation patterns of the neat and doped polylactide samples. ASTM E1131 was used to conduct thermo gravimetric analysis (TGA) of the samples using Pelkin Elmer apparatus. The analysis was conducted from 50 to 600 °C at a rate of 20 °C/min. The results of the TGA study have been tabulated in Table 1.

The TGA Thermograph is as shown in Fig. 1. Although a small dip has been observed in the TGA graph due to loss of moisture and volatile matter, the overall degradation of the

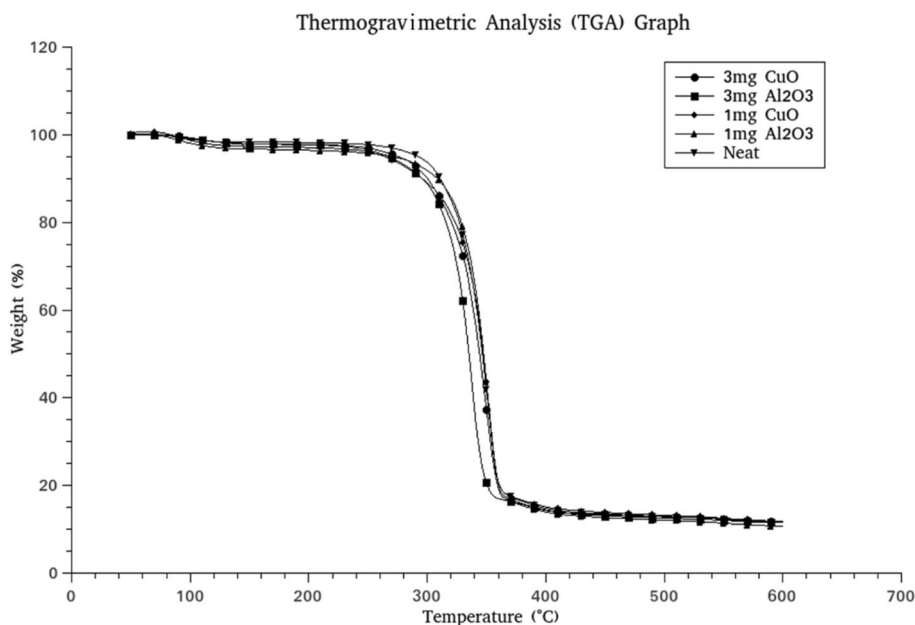
samples may be considered as single stage degradation. The percentage material loss was between 9 and 11% which is in agreement with the work in the literature (Nicolae et al. 2008). It was seen that sample doped with 1 mg nano cupric oxide started thermally degrading prior to all the other samples at 223.3 °C, indicating its low thermal stability. The sample doped with 1 mg nano aluminium oxide however was found to be thermally stable in contrast to all the other samples. The samples doped with nano cupric oxide concluded thermal degradation at the same temperature with an onset degradation temperature of 223.3 °C for sample doped with 1 mg nano cupric oxide and 236.67 °C for sample doped with 3 mg nano cupric oxide.

It should be observed that both the samples doped with nano cupric oxide degraded at the same rate of 8.8889%wt/°C. The addition of nano cupric oxide has caused polylactide to degrade at a slower rate when compared to the neat sample. However the sample doped with 1 mg nano cupric oxide possesses a wide temperature range during the degradation process. The sample doped with 1 mg nano aluminium oxide began degrading at a very later stage at

**Table 1** TGA test measurements

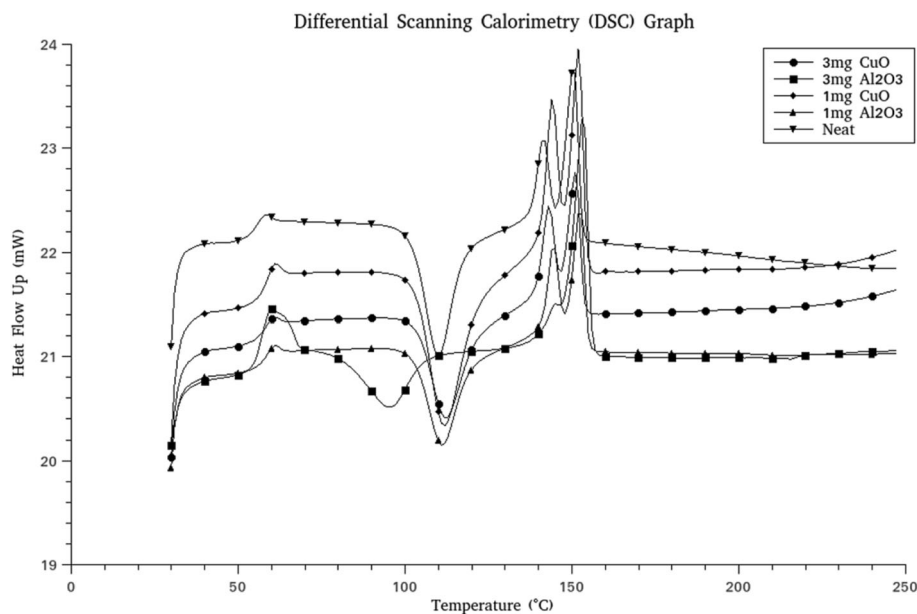
Sl. no.	Sample name	Onset (°C)	Endset (°C)	Rate of decomposition (% wt/°C)	Percentage leftover at 600 °C (%)
1.	Neat	240	361.66	11.4285	11.63
2.	1 mg Al <sub>2</sub> O <sub>3</sub>	250	361.66	11.4285	10.638
3.	3 mg Al <sub>2</sub> O <sub>3</sub>	230	350	13.3333	11.498
4.	1 mg CuO	223.3	363.33	8.8889	11.521
5.	3 mg CuO	236.67	363.33	8.8889	11.571

**Fig. 1** TGA graph



**Table 2** DSC test measurements

Sl. no.	Sample name	Crystallization				Melting				$C_p$ (J/g °C)	% $X_c$
		Onset (°C)	$T_g$ (°C)	Endset (°C)	$\Delta H_c$ (J/g)	Onset (°C)	$T_m$ (°C)	Endset (°C)	$\Delta H_m$ (J/g)		
1.	Neat	53.83	54.54	57.68	16.5392	146.72	150.48	154.08	-14.4176	0.304	33.29
2.	1 mg $Al_2O_3$	55.93	57.82	59.54	15.7682	147.99	152.49	154.89	-11.6909	0.220	29.53
3.	3 mg $Al_2O_3$	52.78	55.67	56.39	20.2709	145.54	151.65	153.07	-18.8773	0.209	42.09
4.	1 mg CuO	56.01	57.69	57.54	24.2727	147.31	151.96	154.46	-20.3423	0.374	47.97
5.	3 mg CuO	55.22	57.22	59.28	13.8673	146.17	150.98	153.66	-14.7481	0.244	30.77

**Fig. 2** DSC graph

250 °C with the smallest temperature range for degradation, leaving the least amount of sample at 600 °C. The sample doped with 3 mg nano aluminium oxide degraded at the fastest rate with 13.3333%wt/°C, being the last sample to completely degrade. Also, the neat polylactide sample left over the highest amount of the sample at 600 °C. The rate of decomposition was significantly effected in the samples doped with 3 mg nano aluminium oxide and in both doping concentrations of nano cupric oxide. However the rate of thermal decomposition is slower when compared to polylactide samples doped with organic dopants, since organic dopants tended to accelerate the rate of thermal degradation of polylactide. Also, the temperature range between the onset and the endset of the thermal degradation was lower in case of organic dopants in comparison with our study (Liu et al. 2012). Degradation at faster rate for 3 mg nano aluminium oxide doped sample and at slower rates for nano cupric oxide samples is clearly evident from the cascading of their respective curves in the TGA thermograph.

### Differential Scanning Calorimetry (DSC)

DSC measurements were carried out to determine the glass transition temperature ( $T_g$ ) and melting peak ( $T_m$ ) using a Pelkin Elmer apparatus following ASTM D3418. All measurements were made at a heating rate of 10 °C/min over a temperature range of 30–250 °C in a dry nitrogen environment. The results of the test have been tabulated in Table 2. The enthalpy of crystallization on heating  $\Delta H_c$  and melting enthalpy  $\Delta H_m$  were measured. The standard formula as shown below was used to calculate degree of crystallinity of the samples.

$$\%X_c = \frac{(\Delta H_m - \Delta H_c)}{\Delta H_f} \times 100,$$

where  $\Delta H_f$  is the theoretical heat of fusion of 100% crystalline polylactide (93 J/g) (Fischer et al. 1973).

Figure 2 shows the graph for DSC analysis of the neat and doped polymer samples. The DSC results indicated an exothermic trough in the temperature range of 80–140 °C which is in agreement with a previous study (Tábi et al.

2010). It was observed that the polylactide sample doped with 3 mg of nano aluminium oxide began to crystallize at about 52.78 °C and concluded crystallization at 56.39 °C. Both these events occurred earlier in comparison with other samples. The sample which began crystallization at the very end was the one doped with 1 mg nano cupric oxide began crystallizing at 56.01 °C and its crystallization process was terminated at 57.54 °C. The sample doped with 3 mg nano cupric oxide recorded highest end set crystallization temperature of 59.28 °C. The neat polylactide sample had a crystallization onset of 53.58 °C and an endset of 57.68 °C. Sample doped with 1 mg aluminium oxide observed highest glass transition temperature of 57.82 °C, lowest glass transition temperature of 54.54 °C was recorded by 3 mg nano aluminium oxide sample whereas neat sample had a glass transition temperature of 55.67 °C.

It was observed that heating the samples above 200 °C did not cause any significant changes in the crystallization patterns of the samples as demonstrated in the research work by Day et al. (2006). Early melting was demonstrated by the sample doped with 3 mg nano aluminium oxide having an onset of 145.54 °C followed by a melting temperature of 150.48 °C and an endset of 153.07 °C. The neat sample exhibited a melting onset of 146.72 °C with a peak at 151.65 °C and an endset of 154.08 °C. However, polylactide sample doped with 1 mg nano Aluminium oxide had the highest range on melting temperatures with an onset of 147.99 °C, peak at 152.49 °C and an endset of 154.89 °C. Heat capacity ( $C_p$ ) of the neat sample is found to be the highest whereas the polylactide samples doped with nano aluminium oxide were observed to have the lowest heat capacity.

Results similar to that in our study have been observed for properties like melting temperature and percentage crystallinity of polylactide doped with nano titanium dioxide. However, the addition of nano titanium dioxide resulted in increase in crystallization temperature and decrease in glass transition temperature (Farhoodi et al. 2012).

### I–V Characterization

The I–V characterization was conducted using a two point probe testing method and Keithley 236 source measure unit. The tests were conducted at the Microtron Center, Mangalore University. The voltage was applied from 0 to 100 V and the corresponding current values were noted. From the obtained values, the I–V graph was plotted. The resistance of the samples was calculated using the slope of the corresponding graphs. The resistivity ( $\rho$ ) of the samples was calculated using the standard formula.

$$\rho = R \frac{A}{t}$$

where R is the resistance of the sample, A is the surface area of the sample and t is the thickness of the sample.

The I–V characteristics data is as shown in Table 3 and Fig. 3. From the I–V characteristics of PLA and its nano composites, it is evident that addition of nano aluminium oxide and nano cupric oxide has reduced the resistivity of the neat polymer by 35–45%. The highest dip in the value of resistivity is observed when PLA is doped with 1 mg nano aluminium oxide since nano aluminium oxide is a high-conductivity filler material (Plesa et al. 2016). It is noteworthy to observe that the increase in the nano aluminium oxide concentration increased the resistance of the nano composite whereas increase in nano aluminium oxide concentration decreased the resistivity of the nano composite. The resistivity values of the nano composites in this study correspond to those of semiconducting polymers ranging from  $10^{10}$  to  $10^{-2} \Omega \text{ m}$  (Rembaum 1970). From these results, we may intuit that PLA may be converted into a semiconducting polymer if doped with nano beryllium oxide and also be converted into a conducting polymer if doped with nano diamond particles.

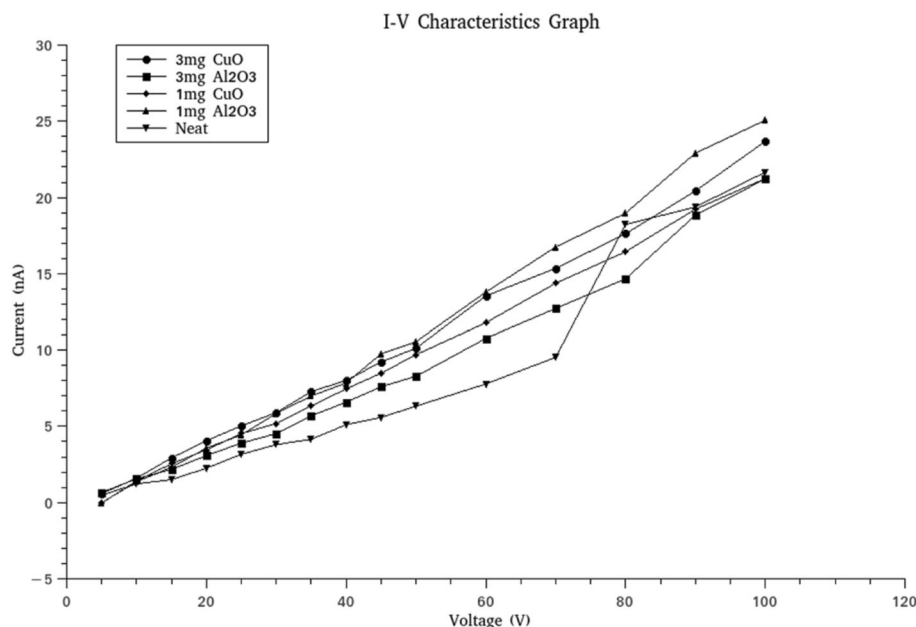
### Conclusion

1. Polylactide and its nanocomposites were sensitive to thermal treatment.

**Table 3** I–V characterisation values

Sl. no.	Sample name	Thickness t ( $\mu\text{m}$ )	Sample area A ( $\text{m}^2$ )	Resistance R ( $\Omega$ )	Resistivity $\rho$ ( $\Omega \text{ m}$ )
1.	Neat	8.57	$2.5 \times 10^{-5}$	$7.1868 \times 10^9$	$2.096 \times 10^{10}$
2.	1 mg $\text{Al}_2\text{O}_3$	10.08	$2.5 \times 10^{-5}$	$3.9768 \times 10^9$	$0.9863 \times 10^{10}$
3.	3 mg $\text{Al}_2\text{O}_3$	9.07	$2.5 \times 10^{-5}$	$4.6623 \times 10^9$	$1.285 \times 10^{10}$
4.	1 mg CuO	10.85	$2.5 \times 10^{-5}$	$5.3579 \times 10^9$	$1.328 \times 10^{10}$
5.	3 mg CuO	11.1	$2.5 \times 10^{-5}$	$4.4021 \times 10^9$	$0.9914 \times 10^{10}$

**Fig. 3** I–V characteristics graph



- Addition of nano aluminium oxide and nano cupric oxide has led to the increase in the thermal stability of the sample doped with 1 mg nano aluminium oxide and decrease in the thermal stability of all the other doped samples.
- Neat polylactide sample had the highest percentage leftover at 600 °C and the sample doped with 1 mg aluminium oxide had the least percentage leftover at 600 °C.
- Sample doped with 3 mg aluminium oxide had the fastest degradation rates whereas those with nano cupric oxide dopants degraded slowly.
- Sample doped with 1 mg nano aluminium oxide was found to be thermally stable in contrast to other doped samples and can be considered as a more suitable material for semiconducting polymer applications.
- Addition of nano aluminium oxide and nano cupric oxide increased the glass transition temperature and melting temperature of neat polylactide.
- Degree of crystallinity of all the doped samples increased with respect to the neat sample except for that of the sample doped with 1 mg nano aluminium oxide.
- Addition of nano aluminium oxide and nano cupric oxide to PLA reduced the resistivity of the samples by 35–45%.

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#### Compliance with Ethical Standards

**Conflict of interest** The authors declare that there are no conflicts of interest.

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