

# Effect of Gamma- Irradiation on Structure, Morphology and Thermal Properties of Novel Polyamide Based Thermoset Obtained by Double Cycloaddition

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Received: 30 April 2020 / Revised: 16 June 2020 / Accepted: 27 June 2020 / Published: 05 July 2020

## ABSTRACT

Cycloaddition reactions gained prominence in macromolecular chemistry for generating macromolecules because of high yields of these reactions, which is a key tool that drives polyaddition reactions. Cycloaddition reaction plays major role in extension of polymerisation or in other words high conversions of monomers to macromolecules of high molecular weights. Until the late 1990s, the major studies regarding cycloadditions in novel polymer synthesis were related to polyaddition reactions. Since then in the field of polymer synthesis the affirmative strengths of these cycloaddition reactions have been exhibited in multi fold polymer design and headway material architecture. Future demand exists in unlatching the capacity of these novel synthetic routes for advanced applications in catalysis, separation, optoelectronics, and analytical media. Thus, we have developed an able and productive synthetic podium for the preparation of a new class of polyimide based on the double 1, 3-dipolar cycloaddition of thiasydnone with bis-maleimide. This paper reports the effect of gamma irradiation on the changes in physico-chemical properties of the polyamide based thermoset synthesised by double cycloaddition approach. The thermoset synthesised by this exclusive approach were irradiated with gamma doses in the range 10- 300 kGy. The substantial effect of gamma radiation and the structural modifications induced on the thermoset have been studied as a function of dose using different characterization techniques such as Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), differential scanning calorimetry- thermo gravimetric analysis (DSC-TGA), Field Emission Scanning Electron Microscope (FESEM) and UV-Vis Spectroscopy.

**Keywords:** Thiasydnone, double cycloaddition, gamma irradiation, thermoset.

## 1 Introduction

The chemistry of mesoionic compounds are booming detailed in the literature [1]. Sydrones, thiasydrones, munchnones and nitrones mainly comes under this category. Among these mesoionic ring systems, sydrones and their functionalised ancestrals have secured much of magnetism because of their wide variety of synthetic and pharmacological applications [2-4]. They have been investigated comprehensively as possible dipoles and doping polymers [5]. From last few years, there is an immense concentration on polymeric materials, which have multi sided

application due to their enormous adaptability, highly tailored production and cost credibility, and it is useful in multiple areas such as engineering technology, defence supplies, and medical devices [6]. Aerospace and microelectronic production corporations essentially demands the advancement of featherweight macro molecules with admirable and exceptional chemical, thermal, and mechanical toughness and durability [7] to ensure proper use of polymers or macro molecules in applications involving exposure to ionizing radiation.

The irradiation of polymeric materials using ionizing radiation is one of the encouraging approaches because of its high penetration proficiency, for modifying or deforming the polymeric materials, like chemical, structural, optical, surface and mechanical properties [8-10]. The synergic effect of radiation with polymeric materials stimulates alterations in the material structure, which causes formation or disappearance of chemical bonds, the production of free radicals, chain scission, cross-linking, chain aggregation and release of hydrogen gas [11-13]. Sometimes in the presence of oxygen, oxidation process starts and oxygen interacts with the free radicals and generates oxidised products [14]. Usually, oxidation and degradation increases with increasing irradiation dose. Different polymers have different reverberations to radiation, which are fundamentally related to the chemical structures of the polymers. As a reflection of this, physico-chemical characteristics of the polymer gets modified [15-20]. By these observations, the development of polymeric material that can with stand high dose of gamma irradiation without change in their intrinsic properties still remains as a major challenge.

The versatile structural characteristics and properties of mesoionic compounds have gained the attention of many researchers to the field of photonics and non-linear optical studies [21–24] and medicinal chemistry [25–31]. Admitting the fact that meso-ionic compounds are well explored in the field of non-linear optical studies and medicinal chemistry, these are less explored in the field of polymer synthesis. The 1,3-dipolar cycloaddition reaction of sydnone to olefinic double bond is well established to produce firstly an unstable 1:1 primary additional product, which in later stage of the reaction eliminates carbon dioxide and undergoes a 1,3-proton shift to become a pyrazoline or eliminates neutral molecule to form a pyrazole system as a final solid product. This bis additional product is due to double cycloaddition of sydnone to two molecules of the olefin after elimination of carbon dioxide. Even though this kind of double cycloaddition reaction is reported with certain

olefins and bis maleimides with sydnones and only a finite number of non aromatic thermosets being prepared using azide–alkyne click chemistry [32-33], no reports were available till date on the synthesis of polymer by double cycloaddition reaction of thiasydnone with bis-maleimides. By these observations and in continuation of our work [34-35] we report herein our attempts for the development of a polymeric system that can with stand a high gamma irradiation by the double cycloaddition of thiasydnone with bis-maleimide.

So, in the present work we subjected thiasydnone, a mesoionic compound with 1,1'-(methylenedi-4,1-phenylene) bismaleimide for double cycloaddition and synthesized a novel macro molecule. The novel polymer thus synthesized was characterised by spectral and microscopic techniques. The novel polymer was irradiated with gamma rays at a dose range of 10-300kGys. The stability of polymer on exposure to ionizing radiation has been studied extensively and several attempts to correlate relative stability with chemical structure of polymers upon irradiation have been made and ensured that this polymer could fit were applications involving exposure to ionizing radiation takes the first priority.

## 2 Materials and Methods

Bis- maleimides were procured from Sigma-Aldrich and used as such without further purification. The infrared spectra of the pristine and irradiated polymer samples were recorded on a Bruker Alpha, Fourier-transform infrared (FTIR) spectrophotometer (Germany). Thermogravimetric analysis of the pristine and irradiated polymers was carried out using Shimadzu DTG-60(Japan) thermo gravimetric analyzer. Differential Scanning Calorimetry was performed in the temperature range from room temperature up to 700 °C at a heating rate 10 °C/ min, for both pristine and irradiated representatives. The nature and morphology of the pristine and irradiated polymers were studied by the Field Emission Scanning Electron Microscope (FESEM) connected with Energy Dispersive Spectroscopy technique (FESEM Ultra 55 Karl-Zeiss).

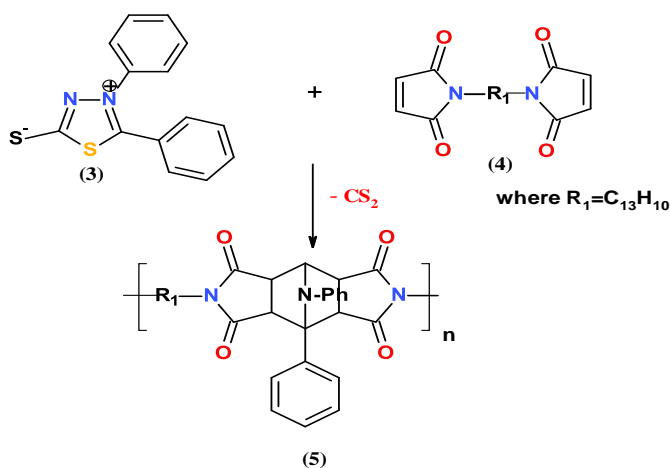
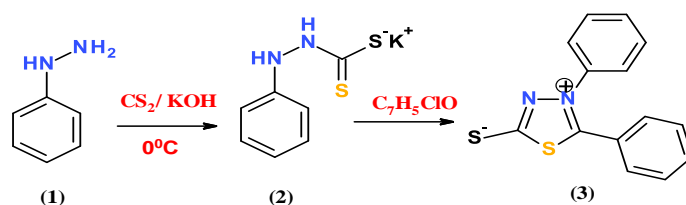
UV/Vis spectroscopy measurements were conducted by UV/Vis Spectrophotometer (Model Tomos UV-1800) in the wavelength range from 800 to 190 nm, in reflection mode. The irradiation was carried out with  $^{60}\text{Co}$  Gamma source of capacity 13455Ci (497.8TBq), at a dose rate of 4kGy/hr at CARRT, (Centre for Application of Radioisotopes and Radiation technology), Mangalore University, Mangalagangothri, India. The samples were irradiated at different absorbed doses 10, 50, 100, 200 and 300kGy, under similar condition.

## 2.1 Procedure for the preparation of polymer

Potassium hydroxide (1.035g, 0.0184mol) was taken in a round bottom flask in 10mL of alcohol and phenyl hydrazine **(1)** (1.0g, 0.00924mol) was added portion wise and the contents were stirred for 30 minutes. Then the reaction mixture was cooled to 0 ° C and added  $\text{CS}_2$  (0.35mL, 0.00462mol) drop wise until yellow coloured solid of potassium  $\beta$ - phenyl dithiocarbazate was formed. The potassium  $\beta$ - phenyl dithiocarbazate was filtered and washed with several batches of diethyl ether until a pure white coloured amorphous powder of potassium  $\beta$ - phenyl dithiocarbazate was obtained. This Potassium  $\beta$ - phenyl dithiocarbazate **(2)** (1.87g, 0.00925mol) was dissolved in 25mL of water and the contents were stirred at 0 ° C. To this solution, benzoyl chloride (1.55mL, 0.011mol) was added drop wise. Care was taken to maintain the temperature in this range. After 3 hours, the yellow precipitate of thia sydnone **(3)** formed was collected by filtration and recrystallized from ethanol to get the pure thia sydnone **(3)** (**Scheme-1**). This thiasydnone (2.50g, 0.00922mol) and 1,1'-(methylenedi-4,1-phenylene) bismaleimide (3.305g, 0.00922mol) were taken in 20mL of *N,N*-dimethylformamide in a RB flask and were stirred under nitrogen atmosphere at 160°C. Carbon disulphide started to evolve at 60°C and was vented through a micro bubbler. Progress of the reaction was monitored by TLC. Once the reaction was complete, the hot solution was poured with care into crushed ice. The polyimide solidified was washed using water and then with

acetone and dried. A brown coloured polyimide was obtained in good yield.

## 2.2 Reaction scheme



## 3 Result and Discussions

For synthesizing polymer, the synthetic methodology we used was double cycloaddition reaction technique between thiasydnone and bis-maleimide. Several reactions, such as Cu(I)-catalyzed azidealkyne reaction, tetrazine-norbornene reaction, Diels-Alder cycloaddition reaction showcases similar features and have been finding applications in fabrication. Among them, the Cu(I)-catalyzed alkyne-azide 1,3-dipolar cycloaddition (CuACC) is one of the preferred techniques. CuACC has been successfully found application in many fields of materials chemistry [36-37]. But the major problem is the toxicity of leftover Copper and selectivity in some cases. This brings a potential limitation of the CuACC reaction when it is used to synthesize or functionalize materials for different applications especially biomedical applications [38-39]. Cycloaddition reactions are a class of reactions that possess the versatile features like high selectivity, excellent yield, rapid

reaction and zero side reactions [40-42]. But the major challenge in macromolecular synthesis is to use equimolar amounts of the building blocks. In addition to this, a simple large-scale purification process and a reasonable timescale and require no tedious fine-tuning of reaction conditions. So here we used most appreciated double cycloaddition reaction for the synthesis.

### 3.1 FT-IR Analysis

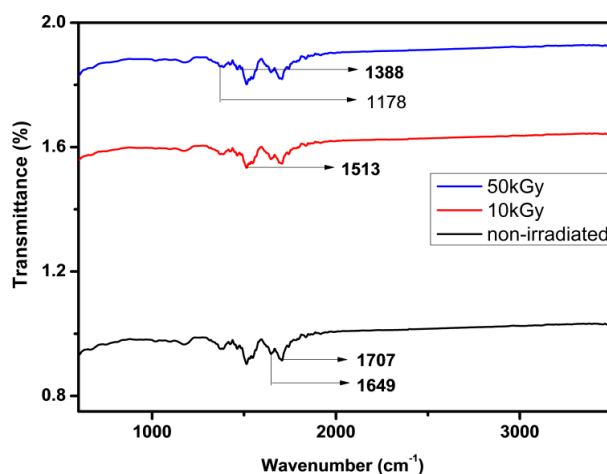
The structure of the newly synthesized polymer was characterized by recording the IR spectra. For polymer sample **5**, a strong absorption band observed in the region of  $\sim 1649\text{ cm}^{-1}$  and  $\sim 1707\text{ cm}^{-1}$  corresponds to the stretching vibrations of two amide carbonyl groups. The  $\text{C}=\text{C}$ -stretching bands were observed at  $\sim 1514\text{ cm}^{-1}$ . The  $\text{C}-\text{H}$  bending band was observed at  $1386\text{ cm}^{-1}$ . The  $\text{C}-\text{N}$  bending band was observed at  $1171\text{ cm}^{-1}$ . The absorption band values corresponding to each infrared vibrations of polymers irradiated with different doses are given in **Table-1**.

**Table 1:** IR absorption bands corresponding to the functional groups of the polymer **5**.

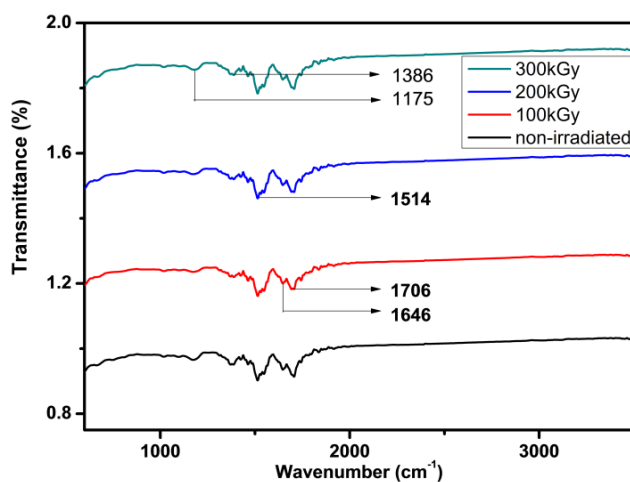
Absorption band ( $\text{cm}^{-1}$ )					Functional group
Polymer sample <b>5</b>					
10kGy	50kGy	100kGy	200kGy	300kGy	
1708,1647	1707,1647	1706,1646	1708,1647	1707,1647	C=O stretch.
1513	1513	1514	1514	1514	C=C stretch.
1387	1388	1388	1387	1386	C-H bend.
1172	1175	1173	1172	1175	C-N bend.

The gamma irradiated samples of the polymers showed IR vibrational absorption bands approximately at the same wave numbers as that of pristine sample. The polymer showed no distinguishable changes in structure between non-irradiated representative and those irradiated with particular doses of gamma rays. The stretching vibrations of two amide carbonyl groups for the irradiated polymer samples (10kGy-300kGy) were observed in the region of

$1708\text{ cm}^{-1}$ -  $1706\text{ cm}^{-1}$  and  $1647\text{ cm}^{-1}$ - $1646$ . The  $\text{C}=\text{C}$ - stretching bands for irradiated polymer was observed in the region of  $1514\text{ cm}^{-1}$ - $1513\text{ cm}^{-1}$ . Similarly  $\text{C}-\text{H}$  bending for the irradiated sample was observed in the region of  $1388\text{ cm}^{-1}$ - $1386\text{ cm}^{-1}$ . Even the  $\text{C}-\text{N}$  bending band was observed at the same wave number as that of the pristine. The IR spectra of the polymer irradiated with different doses of gamma radiation are as shown in **Fig. 1** and **Fig. 2**.



**Figure 1:** FT-IR spectra of pristine and polymer (**5**) irradiated with dose of 10kGy and 50kGy

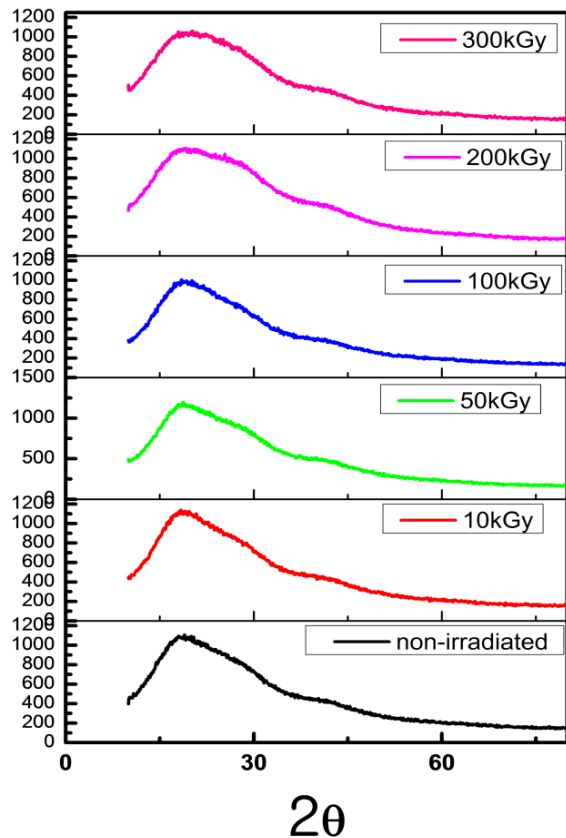


**Figure 2:** FT-IR spectra of pristine and polymer (**5**) irradiated with dose of 100kGy, 200kGy and 300kGy

### 3.2 XRD Analysis

The powder XRD of polymer sample **5** was recorded between  $2\theta$  range  $10^\circ$  -  $90^\circ$  before and after gamma ray irradiation. The pristine showed a halo between  $2\theta$  values of  $10^\circ$ - $35^\circ$  centred at  $18.92^\circ$ . The profile of the halos reveals that the

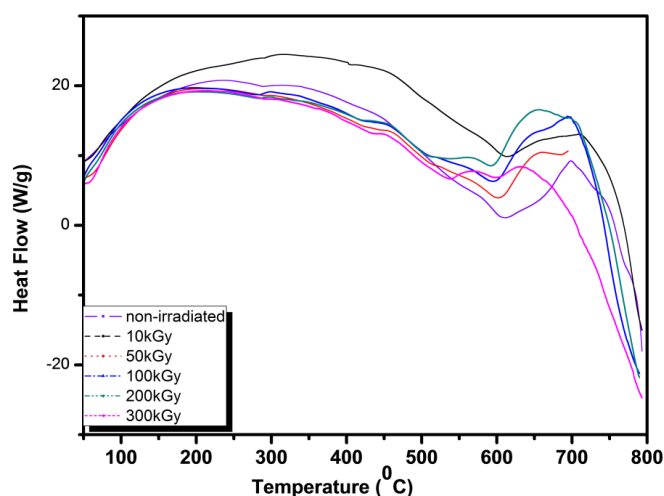
polymer samples are partially crystalline with the leading dominant amorphous phase. However, when the sample was irradiated with gamma ray doses of 10Gy's, there were slight shift in the peak position from  $18.92^\circ$  to  $18.31^\circ$  for 10kGy's and slightly sharpens as seen in **Fig. 3**. This distinct anomaly phenomenon was observed for only irradiation dose of 10kGy's. This may be due to the cross linking of the polymer chains under gamma irradiation leading to the change in short range crystal phases of polymer. This cross linking leads to the re-orientation of polymer chains to the crystalline region. However at higher irradiations (50kGy's and above) the centre of the peaks was shifted to  $18.78^\circ$ ,  $19.00^\circ$ ,  $23^\circ$  and  $23.7^\circ$  for polymer sample irradiated with 50, 100, 200 and 300kGys respectively and seen as slightly broader indicating the amorphous nature as seen and confirmed by **Fig. 3**. This is because, at higher gamma doses the polymer chains would undergo chain scission which predominates over the cross coupling of polymer chains. The XRD pattern for non-irradiated and irradiated polymer sample **5** is shown in **Fig. 3**.



**Figure 3:** The XRD pattern of pristine and irradiated polymer **5**.

### 3.3 DSC- TGA Analysis

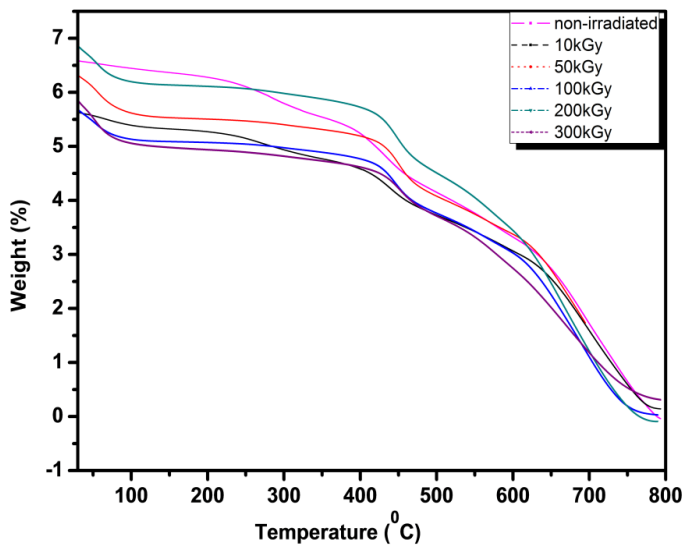
The DSC curves for non-irradiated and irradiated polymer samples **5** is as shown in **Fig. 4**.



**Figure 4:** DSC thermograms for pristine and polymer (**5**) irradiated with different doses gamma rays.

The DSC curves of the pristine and gamma irradiated polymer samples for **5** showed no prominent endothermic peaks corresponding to the glass transition temperature ( $T_g$ ). The pristine sample **5** showed commencement of oxidation of polymer chains at a temperature of  $236^\circ\text{C}$  which further undergo degradation and starts melting above  $600^\circ\text{C}$ . However, the gamma irradiated samples showed a notable increase in resistance to oxidation temperature from  $236^\circ\text{C}$  to  $316.5^\circ\text{C}$  at an irradiation dose of 10kGy. This may be due to the cross linking of the polymer chains under gamma irradiation which causes increase in crystalline phase of the polymer by forming new bonds between the chains as evidenced in XRD. But the trend was not similar at higher irradiation doses of 50, 100, 200 and 300kGy's where the degradation temperature slightly decreased with increase in irradiation dose. This is because at higher gamma doses the polymer chains would undergo scission that reduces the number of entanglements per molecule which predominates over the cross linking. Chain scission can also act to reduce intermolecular stress in the amorphous region, thus increasing chain mobility. The increase in mobility allows some molecules to re ordered. The exothermic peak positions of the pristine and gamma irradiated samples are given

in **Table-2**. The percentage weight loss of the pristine and gamma irradiated samples was calculated with respect to their degradation temperature (stage-I) and up to 600°C (stage-II) and the results are tabulated in **Table-2**. The initial weight loss below 100 °C is due to the loss of moisture content. The onset of major degradation of polymer **5** was observed at 236°C. The weight loss corresponding to this degradation was found to be 5.68 %. The TGA curves for non-irradiated and irradiated polymer samples **5** are shown in **Fig. 5**.



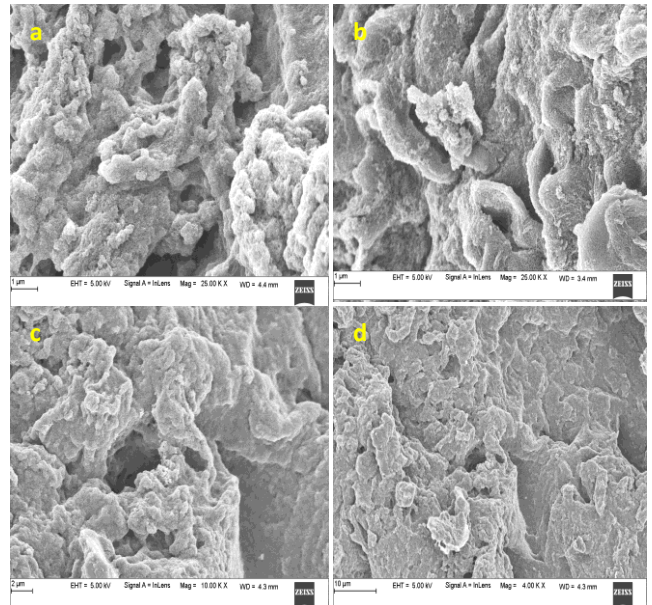
**Figure 5:** TGA curves for pristine and polymer (**5**) irradiated with different doses of gamma rays.

**Table 2:** Percentage weight loss as a function of temperature for pristine and polymer **5** irradiated with different doses of gamma rays.

Sample code- <b>5</b>	Thermal Degradation Temperature (°C)	Weight Loss (%) (Temp. °C)	
		Stage- I (at Degradation temperature)	Stage-II (at 600°C)
Non-irradiated	236.0	5.68	46.12
10kGy	316.5	13.50	37.74
50kGY	308.0	22.55	39.34
100kGY	300.3	12.85	39.69
200kGY	295.8	12.80	43.36
300kGy	289.2	18.34	43.82

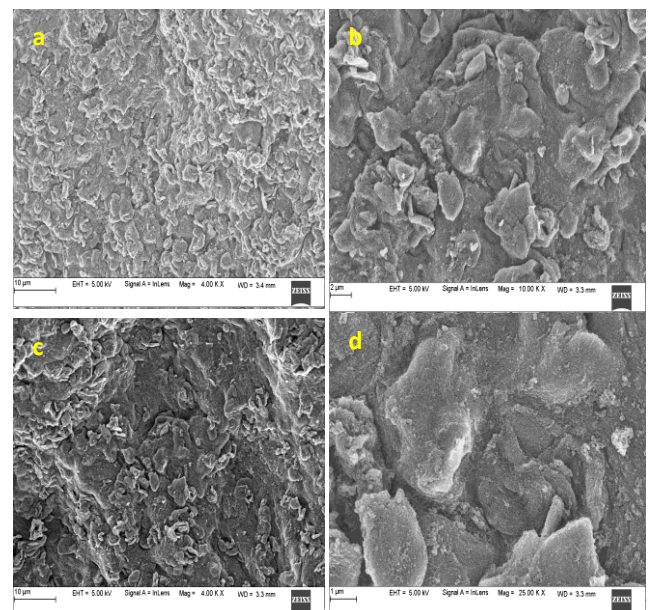
### 3.4 SEM Analysis

The SEM micrographs of pristine polymer sample are shown in **Fig. 6**.



**Figure 6:** SEM micrographs of pristine a) and b) 1µm c) 2µm and d) 10µm

The surface of polymer sample **5** was seen as massive irregular structures indicating the amorphous nature. It showed spongy type appearance distributed unevenly over the entire surface. The morphological changes observed on surface of polymer samples on gamma irradiation are shown in **Fig. 7**.

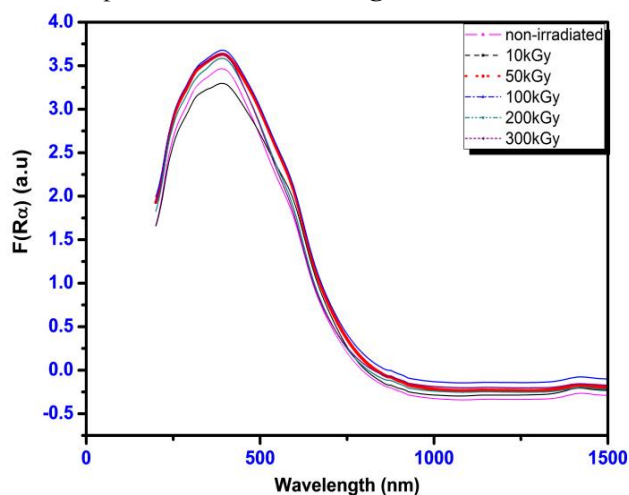


**Figure 7:** SEM micrographs of irradiated polymer **5** with a dose a) 100kGy b) 200kGy c) 300kGy and d) 300kGy.

The surface of the sample irradiated with 100kGy was roughened and appeared darker a bit than the pristine and there is no much change in irregularities as seen in image **a** of **Fig. 7**. For sample irradiated with 200kGy, the surface was seen to be darker than the sample irradiated with 100kgy. At higher dose of 300kGy, the irregularities on surface decreased a bit but there was no plain appearance that causes due to the surface charring effect on gamma irradiation.

### 3.5 UV- Visible Spectroscopy

The UV-Visible diffuse reflectance spectra of non-irradiated and irradiated polymer sample **5** was plotted as a corresponding Kubelka Munks function,  $F(R_\infty)$  values versus wavelength of UV-Visible radiation. The diffuse reflectance spectrum of pristine and irradiated polymer samples **5** is as shown in **Fig. 8**.



**Figure 8:** UV-Visible spectra of pristine and polymer (**5**) irradiated with different doses of gamma rays.

The  $\lambda_{\max}$  values corresponding to different irradiation doses are tabulated in **Table-3**.

**Table 3:**  $\lambda_{\max}$  values for pristine and polymer **5** irradiated with different doses of gamma rays.

Gamma Dose (kGY)	$\lambda_{\max}$ (nm)
	<b>5</b>
Pristine	389.90
10	390.00
50	391.15
100	392.34
200	390.60
300	390.20

The diffuse reflectance spectrum of **5** showed  $\lambda_{\max}$  at 389.90 due to the  $\pi$ - $\pi^*$  electronic transitions. The gamma irradiated samples of **5** showed  $\lambda_{\max}$  nearly at the same wavelengths as that of pristine except sample irradiated with 50 and 100kGY indicating that there is no significant affect of gamma irradiation on the energy levels of the synthesized polymers there by indicating good structural stability towards gamma irradiation dose as evidenced in FT-IR. For this particular dose of 50 and 100kGy the  $\lambda_{\max}$  shifted from 389.90 nm to 391 nm and 389.90 nm to 392 nm respectively. This increase may be due to the formation of some electronic levels inside the forbidden gap at this irradiation dose. This shift may also be attributed to irradiation-induced defects in the polymeric material. In addition to this, here in this case after irradiation with gamma rays, broadening of absorption edge is observed. This broadening may be due to the formation of some defects due to gamma irradiation, i.e. the formation of conjugated bonds.

## 4 Conclusions

We reported the synthesis of novel macromolecule by a 1,3-dipolar double cycloaddition process and successfully synthesized light weight and excellent material with high chemical and thermal robustness. It exhibits a facile synthetic access to a polyamide-based family of fully aromatic thermosets. The synthesized polymer was tested for its structural and thermal stability before and after gamma irradiation. The thermoset exhibited an affirmative structural and thermal stability even after high gamma irradiation dose of 300kGy as evidenced by FT-IR and DSC-TGA studies. The availability of alternative possible replacement of monomers demonstrates the embryonic potential of this tuneable material platform as a high-performance matrix for the future developing technologies. Finally, this polymer could fit were applications involving exposure to ionizing radiation takes the first priority.

## 5 Declarations

### 5.1 Acknowledgements

The authors are thankful to the Coordinator, DST- PURSE, USIC and Department of Physics,

Mangalore University, for providing facilities for the characterization of polymers and technical support to carry out the work. Authors also acknowledge DAE-BRNS, Mumbai, for the financial support in the form of major research project.

## 5.2 Study Limitations

There was non- availability of GPC instrument for the determination of molecular weight of the synthesized polymer. Most of the GPC instruments are based on Tetrahydrofuran. Our polymer sample was only partial soluble in THF.

## 5.3 Funding Source

Authors are thankful to DAE-BRNS for the financial support.

## 5.4 Competing Interests

Authors declare that there is no conflict of interest exists.

## How to Cite this Article:

B. Kalluraya, K. B R, and H. M. Somashekarappa, "Effect of Gamma- Irradiation on Structure, Morphology and Thermal Properties of Novel Polyamide Based Thermoset Obtained by Double Cycloaddition", *J. Mod. Mater.*, vol. 7, no. 1, pp. 17-25, Jul. 2020. doi:10.21467/jmm.7.1.17-25

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