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Evaluation of Porosity for Gamma Irradiated Poly (ethylene oxide): A New Approach Using Microscopic Image Aided with Computer Programming

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Evaluation of Porosity for Gamma Irradiated Poly (ethylene oxide): A New Approach Using Microscopic Image Aided with Computer Programming

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***Abstract*—**

Porous films of Poly(ethylene oxide)(PEO) are synthesized by casting using gamma irradiated:(a)PEO powder (S-series) &(b)PEO- solution (L-series). The experimental pore-size and distribution of the films is studied using BET adsorption and reported as a function of irradiation dose with concentration of 2 and 4 wt.%. A computer program [PROGIMAGE-POR] is reported for determination of porosity and pore-distribution using SEM and correlated with BET. Novelty of PROGIMAGE-POR lies in the exposure of newer pore regime in which experimental pores exists as a part. Theoretical analysis based on aerosity (2D-pores) and porosity (3D-pore) gives an estimate of the tortuous path of void space.

***Index Terms*—**

Porosity; Morphology; Pore-size distribution; Computer Program; Grey scale pixel; Gamma Irradiation

I. Introduction

Pores (or voids) are formed in polymer films irrespective of any preparation technique like gel casting, emulsion freeze drying, acylation, solution casting etc. [1]. The basic challenge lies in the optimization of pore-size and porosity distribution based on the purpose of application. In lieu of this, pore phase has recently emerged as an important subject to be well studied and optimized for multifaceted applications. Study of pore phase bears significance in identification of individual grain interfaces through the indirect outcome of the processes at micro-scale also termed as pore-scale by Wildenschild et al. [2]. Study of porosity in terms of quantification and distribution involved multiple well studied tools, viz. adsorption technique (BET technique, nitrogen adsorption) [3], intrusion procedures as mercury porosimetry [4], X-ray tomography etc. [5]. The major disadvantages associated with X-ray tomographic imaging includes artifacts produced by sample rotation and metal mountings, costly instrumentation, long measurement time for single sample and large data files. In the present article, the porous Poly (ethylene oxide), PEO films perturbed using high energy gamma dose (1 kGy-30kGy, in powder and methanol solution state) is subjected to BET (Brunauer–Emmet–Teller) adsorption technique to study pore size distribution (PSD). A novel computer program termed as **$PROG_{IMAGE\text{-}POR}$** is developed for determination of porosity, it's size and distribution and is correlated with the experimental outcomes. The program, $PROG_{IMAGE\text{-}POR}$ is based on the scanning electron microscopic (SEM) images as an input source file. However, the novelty of $PROG_{IMAGE\text{-}POR}$ lies in the exposure of newer or undetected pore regime.

II. Salient Results and Discussion

The basic flow chart for determination of porosity by $PROG_{IMAGE\text{-}POR}$ is shown in **Figure 1**.

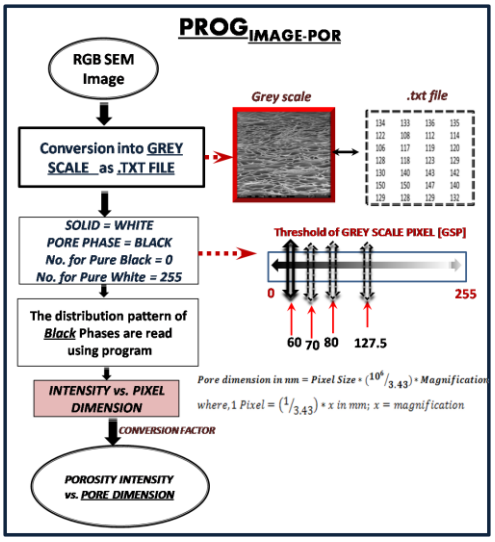

***FIG. 1.** Schematic flow chart for the developed program [$PROG_{IMAGE\text{-}POR}$].*

SEM images of experimental PEO samples (**Table 1** in manuscript) are converted to *8bit type* image using ImageJ software and saved as *text image* with *.txt* file extension. Each of these text images contains integer numbers ranging from 0 to 255 as a 2D array with dimension *width*height* of the SEM image. We have set GSP to a particular threshold value GSP_{th} to signify solid (GSP >GSP_{th}) or void (GSP < GSP_{th}) phase. The outcome of $PROG_{IMAGE\text{-}POR}$ is obtained in the form of "*Pore size distribution [Porosity Intensity vs. Pixel dimension]*" and "*Average porosity [Av. Porosity vs. dose]*". The pixel dimension as obtained from program is converted to pore-dimension using Eq. 1 and 2. Equation 2 is calculated from IMAGEJ software. The average dimension of pores is

found to lie within 100-500 nm for PEO films prepared using irradiated powder (2/4-S series) and/or methanol solution (2/4-L series) (**Figure 2**).

$$Pore\ dimension\ in\ nm = Pixel\ Size * (^{10^6}/_{3.43}) * Magnification$$

$$where, 1\ Pixel = (^{1}/_{3.43}) * x\ in\ mm;\ x = magnification \quad (1\ \&2)$$

Table I: Sample identification of experimental Poly(ethylene oxide) films

Sample Irradiated	Concentration of PEO ($g.ml^{-1}$)	Sample ID					
		Dose (kGy)					
		1	5	10	15	20	30
Powder Irradiation	0.02	2S-1	2S-5	2S-10	2S-15	2S-20	2S-30
	0.04	4S-1	4S-5	4S-10	4S-15	4S-20	4S-30
Solution Irradiation	0.02	2L-1	2L-5	2L-10	2L-15	2L-20	2L-30
	0.04	4L-1	4L-5	4L-10	4L-15	4L-20	4L-30

In addition to the mentioned pore-dimension regime, smaller pores of <100 nm exists for unirradiated PEO matrix which disappear upon subsequent irradiation.

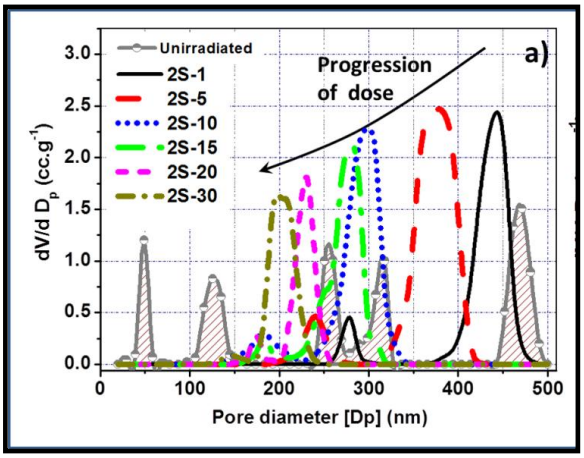

FIG. 2. *Pore-size distribution determined from BET technique for 2S- series as a function of irradiation dose.*

Irrespective of powder or solution state irradiation, predominant contribution of scission increases % porosity sharply till 5 kGy in all the samples. However, with further increase in dose, matrix densification results due to the formation of innumerable scission fragments in S-series. **Figures 3** describes the pore-size distribution obtained from $PROG_{IMAGE\text{-}POR}$ using SEM images for 2S series for 1, 10 and 30 kGy in five magnification regimes from 100x to 10000x respectively. It could be observed that, PSD obtained from the program consists of wide regime of pores from 10^2 to 10^5 nm. It is observed that 50000 x magnifications unveils pore in the dimension of ~ 10^3 nm irrespective of sample type. Lower dose of 1 kGy generates uniform distribution of pore with intermediate intensity. Population of pores is found to increase with dose increment to 10 kGy especially for 2/4S-series. Erratic distributions are obtained with highest dose of 30 kGy which generates smaller pore (less than 10^2 nm) for 2S-series and larger pores (greater than 10^3 nm) for 2L-series. The obtained observations are further supported by morphological studies and aerosity.

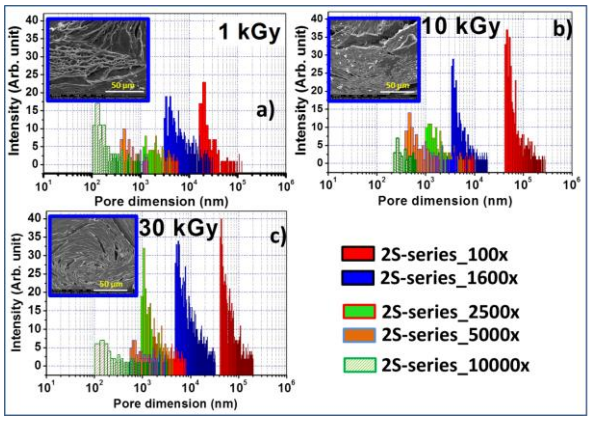

FIG. 3. *Pore-size distribution obtained from $PROG_{IMAGE\text{-}POR}$ as a function of SEM magnification for irradiated 2S-series The inset figures correspond to the SEM micrographs at 2500x magnification.*

III. CONCLUSION

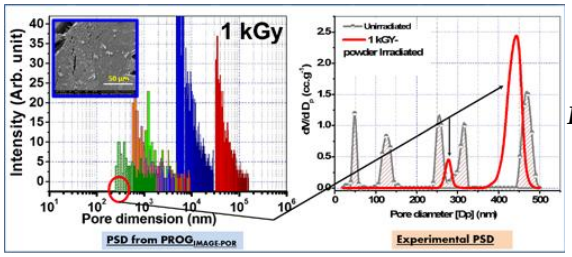

Fig. 4

A new, simple and novel computer program termed as $PROG_{IMAGE\text{-}POR}$ is reported for study of porosity and its distribution of Poly(ethylene oxide) (PEO) films prepared with gamma irradiated [1-30 kGy] powder (2/4S-series) and methanol solution (2/4L-series). It is found that the experimental data related to porous structure of PEO films obtained from BET (100-500 nm) measurements appears to be a part of the entire range of pore spectrum (~ 10^2-10^5 nm) obtained from 2D SEM image based program (**Figure 4**).

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REFERENCES

[1] A.M. Laera, L. Mirenghi, M. Schioppa, C. Nobile, L. Capodiecl, A. G. Scalone, F. Di Benedetto, L. Tapfer. *Mater. Res. Express.* **3**, 085007. (2016).
[2] D. Wildenschild and A. P. Sheppard. *Adv. Water Resour*. 51, 217 (2013).
[3] A. M. Stephan and D. Teeters. Electrochim. Acta. **48**, 213 (2003).
[4] J. L. Calvo, A. Hernandez, P. Pradanos, L. Martibnez and W. R. Bowen. *J. Colloid Interface Sci.* **176**, 467 (1995).
[5] A. Pyun, J.R. Bell, K.H. Won, B.M.Weon, S.K. Seol, J. J. Ho and C. W. Macosko. *Macromolecules.* **40**, 2029 (2007).