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ScienceDirect

Procedia Chemistry 19 (2016) 477 – 484

Procedia Chemistry

5th International Conference on Recent Advances in Materials, Minerals and Environment (RAMM) & 2nd International Postgraduate Conference on Materials, Mineral and Polymer (MAMIP), 4-6 August 2015

Effectiveness of A Simple Image Enhancement Method in Characterizing Polyethylene Foam Morphology using Optical Microscopy

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Abstract

A simple image enhancement technique was proposed to observe and analyze cell structure of polyethylene foams using optical microscopy. Two coating techniques were implemented in this study; one being a simple black soot coating and the other utilized a commercial Au/Pd coating that is commonly used for Scanning Electron Microscopy (SEM) observation. The former technique was forwarded after considering that when the sample is white in color, usually microscopic observation via optical microscopy is quite difficult. Polarized light was also applied as another supporting tool to counter the light reflectance problem occurred in the observed white sample. Good quality digital images were successfully produced using both coating techniques with the assistance of polarized light projected via the optical microscope. It was proven that the sample with the proposed black soot coating is able to give high quality image with high level of contrast. The results showed that images with clear cell boundaries were obtained and these images were comparable or much better than those obtained with the Au/Pd coating technique. Finally, images with improved contrast were inverted using an image analysis procedure which allows better determination of foam cell features in polyethylene foams. The use of the proposed technique as supplementary device for real-time microscopic observation during foam compression was also proven to be a success.

Peer-review under responsibility of School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia

Keywords: Polyethylene foams; optical microscope; black soot; polarized light

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Peer-review under responsibility of School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia
doi:10.1016/j.proche.2016.03.041

1. Introduction

In principle, there are various techniques that can be utilized to characterize material's morphology such as optical microscopy (OM), scanning electron microscopy (SEM), X-ray micro-tomography, etc. However for polymeric foams, several limitations can be encountered in obtaining a good image. In addition, it becomes more complicated to obtain sufficient high contrast especially when OM is used to define cell walls for closed-cell foams with transparent or white color. Technically, it is complicated due to the cell faces of these materials are able to scatter and/or reflect light thus preventing OM from viewing its interior[1]. This is the reason for most researchers to opt to SEM rather than using OM in characterizing the properties of such foams[2].

There are limited reported studies that focused in addressing the contrast problem faced during characterization of these polymeric foams[3,4]. However, techniques proposed in these studies are only suitable for large foam cell of rigid and semi rigid foams and subsequently become difficult to be applied for flexible foam such as elastomeric foams. The principle of most of the techniques is to reduce light reflectance that often occurred in polyolefin foams which highlighted the cell walls making it optically "blocking" the detection of cell boundaries.

However, in this study, the utilization of optical microscope was preferred due to the motivation in giving greater freedom to researchers in conducting microscopic observation without having to rely heavily on expensive instrument such as SEM. In many developing countries, specialized equipments such as SEM would be a luxury. Even in countries like Malaysia, although the number such facilities are increasing; several drawbacks would create obstacles that might hold back the progress of research activities. Problems such as frequent breakdowns, high maintenance cost, and increasing number of researchers having the same needs for such instrument are some common difficulties faced by researchers in this part of the world. Therefore, the utilization of optical microscopy was proposed to give better solution to these problems thus giving more autonomy to the researchers to dictate the pace of their research activities. In addition, the use of optical microscope for microscopic observation is far more economical and simpler compared to SEM. Optical microscopy is also considered to be a quick, non-contact and non-invasive technique which offers new approaches in fulfilling its requirements besides now having extra features not found in expensive microscopy techniques; e.g. device portability[5].

In view of the abovementioned advantages, there are still a few old problems in utilizing OM in certain microscopic observation. One of them is the inability of OM to create contrasting micrographs if the observation involves a sample that is white in color and having complex morphology such found in polyethylene foams. Due to the transparency of the foam cell walls, the OM images tend to have higher glare occurrence due to multiple light scattering and reflectance of the optical light[1] by the cell walls making it impossible to detect the cell boundaries. The key of solving this glare problem is to reduce the reflection and scattering of light and in this study, a simple glare reduction technique was proposed and tested. The proposed technique was benchmarked with a commercial gold/palladium (Au/Pd) coating technique which is well-known able to give better contrast in Scanning Electron Microscopy (SEM) technique[6,7]. The feasibility of the proposed technique to be used as a supplementary visualization device during real-time monitoring of foam compression was also evaluated.

2. Experimental

2.1 Sample Preparation

Samples were cut into the dimension of approximately 10 x 10 x 5 mm for image analysis purpose. A standard cutting procedure was established to ensure that flat surfaces were obtained in order to acquire good digital images. The samples were cut using sharp shaver blades to avoid implicating cell collapse and the cutting direction was perpendicular to the foam rise direction. The cut foam samples were then subjected to two coating techniques to compare the proposed black soot coating technique with a commercial sputter coating technique.

The first coating technique involved exposing the sample to black soot created from combustion of a kerosene lamp having a setup shown in Fig.1. The effective distance of the samples from the lamp was established (250 mm) after conducting several trial runs and is critical since it can affect the quality of the coating technique. The incomplete combustion of the lamp gave out black smoke consisting suspended fine carbon particles that can attach evenly onto the samples surfaces[8]. The principle of this technique is to introduce a layer of carbon coating that can reduce the light reflectance and scattering within the foam cell walls thus solving the glare problem giving better contrast.

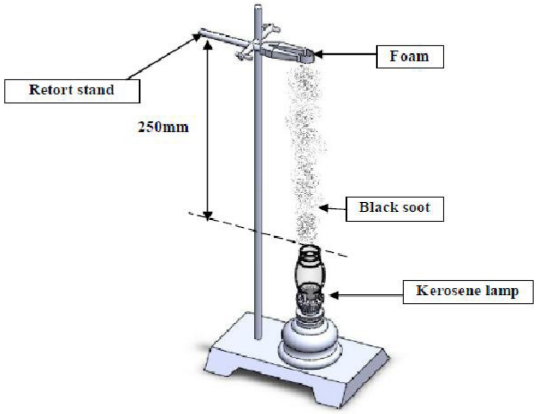

Fig. 1. Black soot coating technique setup.

The second technique utilized a commercial sputter coating, BioRad SEM Coating System. The samples were attached to the mounting fixture with electrically conductive carbon tape and placed in vacuum chamber having a minimum pressure of 2 mbar for 60 seconds. Sample from this coating technique was then submitted to scanning electron microscopy (FESEM Zeiss Supra 35VP-25-58). The SEM analysis was implemented to establish cell size and cell distribution of the prepared sample so that it can be used as a benchmark to the measurement done using optical microscopy.

2.2 Image capture using mini portable digital microscope

A portable digital microscope (Dino-lite AM413ZT) was utilized in this study to observe the foam structure. This procedure was conducted on samples exposed to both black soot and Au/Pd coating technique. A few locations were captured in parallel lines as shown in Fig.2(a) (labelled i, ii, iii and iv) and multiple-image combination were applied to generate picture wider perspective of the sample as shown in Fig.2(b).

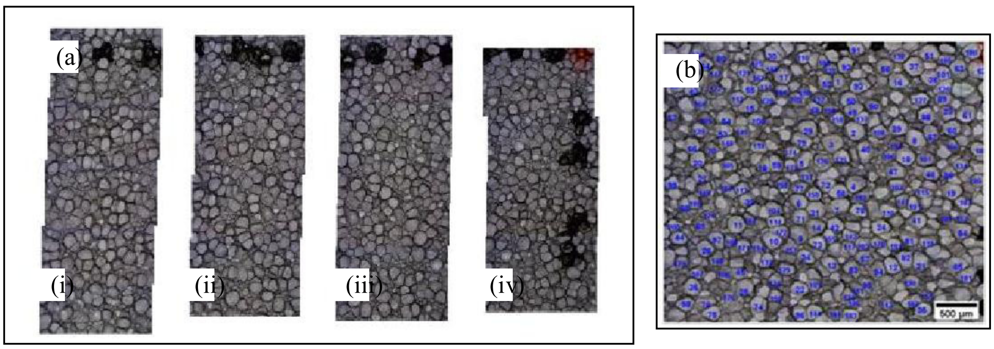

Fig. 2. The microstructure of LDPE foam cells captured by the mini portable digital microscope, (a) before; (b) after image combination.

Image analysis software (Image J) was used to analyze cell size and cell size distribution in the captured images. Several locations of each sample were measured and accumulated to achieve a good description of the cell size distribution. At least 200 cells were measured in the assembled image to achieve statistical accuracy and an example of cell size distribution result is given in Fig.3. It is clear from the figure that cell size is in the vicinity of 235-255 µm and has monodisperse distribution. The utilization of image analysis technique with the assistance of the proposed black soot coating technique, gave the opportunity for the optical microscopy setup to conduct better quantitative microstructural analysis.

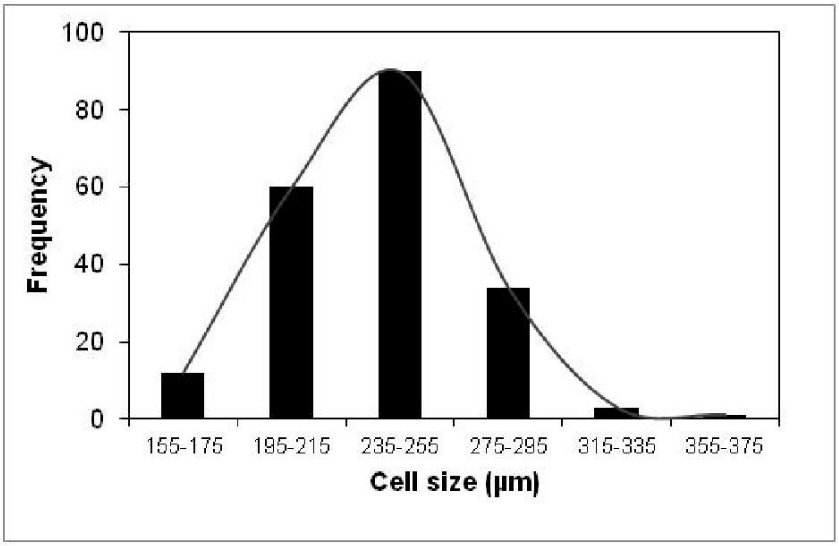

Fig. 3. Cell size distribution of PE foam cell obtained using black soot coating and polarized optical microscope.

2.3 Compression test verification

Samples were prepared by cutting the foam block into cube 25 x 25 x 20 mm dimension. Five samples were tested using universal testing machine model Testometric Micro 500 UTM machine (Rochdale, England). A crosshead speed of 10 mm/min was used in accordance with ASTM D3575. It is also important to note that all the samples were compressed in the foam rise direction. Samples were deflected up to 70% compression strains.

3. Results and Discussion

3.1 Foam Cell Image Quality

The utilization of coating in this study is a supplementary technique which allows observation of optically-challenged PE foam structure to be conducted using optical microscopy. Fig.4 shows the effect of coating technique (a) black soot coating (b) Au/Pd coating on the sample images captured by the portable digital microscope. It is clear from the figures that both coating techniques exhibit high quality images under polarized light compared to those obtained under un-polarized light. Images captured under un-polarized display relatively blurred images and it is apparent that the boundaries of the cells are difficult to be differentiated. However when the polarized light was utilized to observe the coated foam surface, high quality images were obtained where the boundaries of each cell can be clearly visualized.

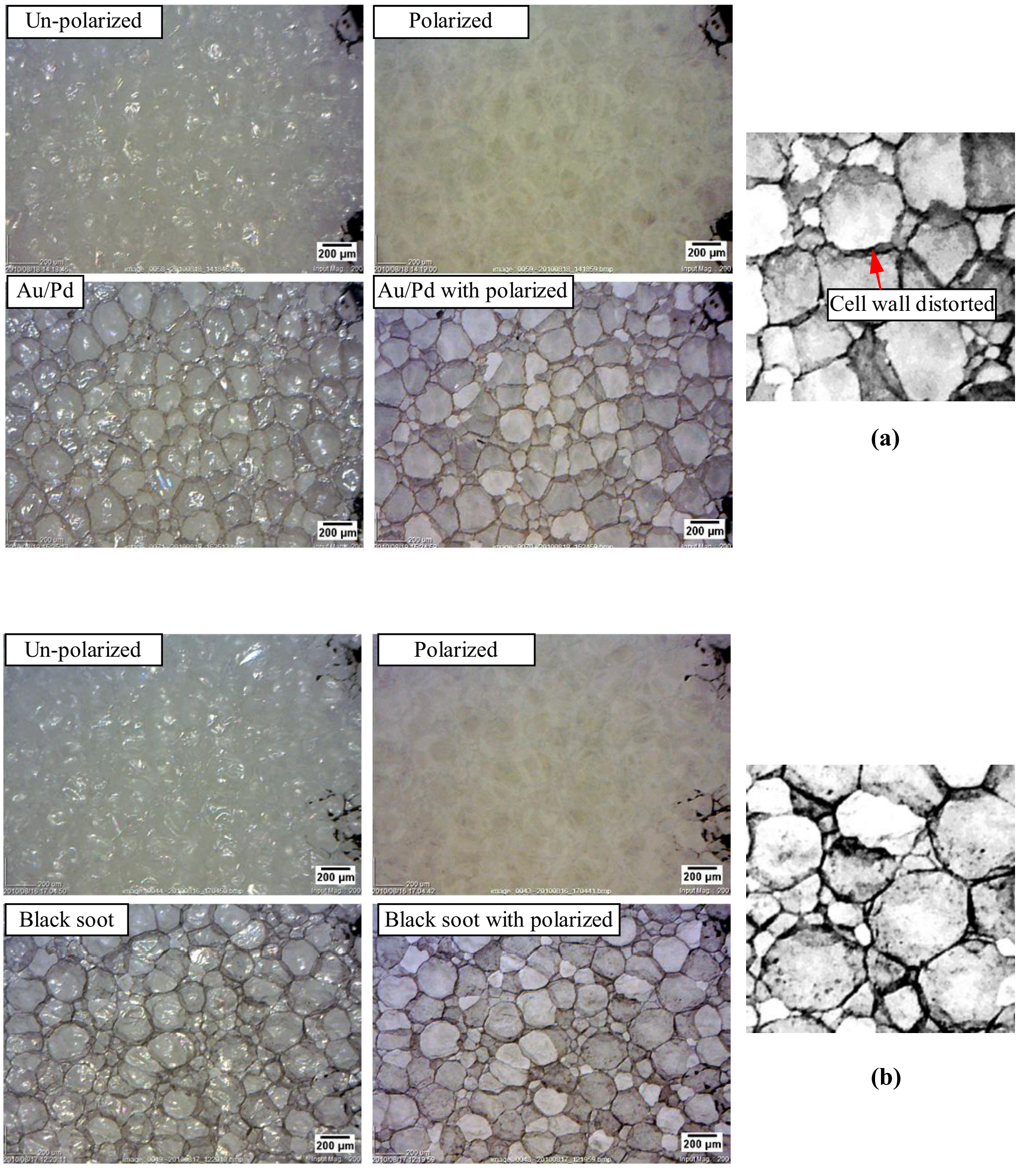

Fig. 4. Optical microscopy micrograph of the closed-cell PE foams with different coating techniques (a) Au/Pd ;(b) Black soot.

It can be observed that both coating substances (i.e. black soot and Au/Pd) have spread out quite evenly onto the surfaces and therefore, able to reduce the light scattering and enhance the image contrast. The light reflectance in the sample has been reduced by the coating materials and subsequently produced good image quality. From the figure, cell boundaries can be clearly differentiated with the cell walls appear to have thicker coating and the empty spaces or cavities (which accommodate the gas phase), are displaying lighter appearances.

If both images from optical microscopy and SEM were compared, sample with Au/Pd coating have more diffused and relatively blurred cell boundaries. This is due to the cell walls were seem to be distorted when the sample surface were exposed to Au/Pd coating procedure. This can be associated with heat generated during sputtering process where high energy ions were bombarded to the thinner cell walls. It is projected that if the foam sample has thicker cell walls, a better image can be obtained with the Au/Pd coating technique. As for the sample coated with black soot, the captured image was relatively better where distinctive and sharp edges of cell walls can be detected throughout the selected observation area.

3.2 Comparative Cell Size Distribution

Comparison of cell size distribution results between SEM and OM (with improved image enhancement technique using black soot coating) are displayed in Fig. 5. Both techniques showed the same trend which proved that measurement made with OM is as reliable as the SEM measurement.

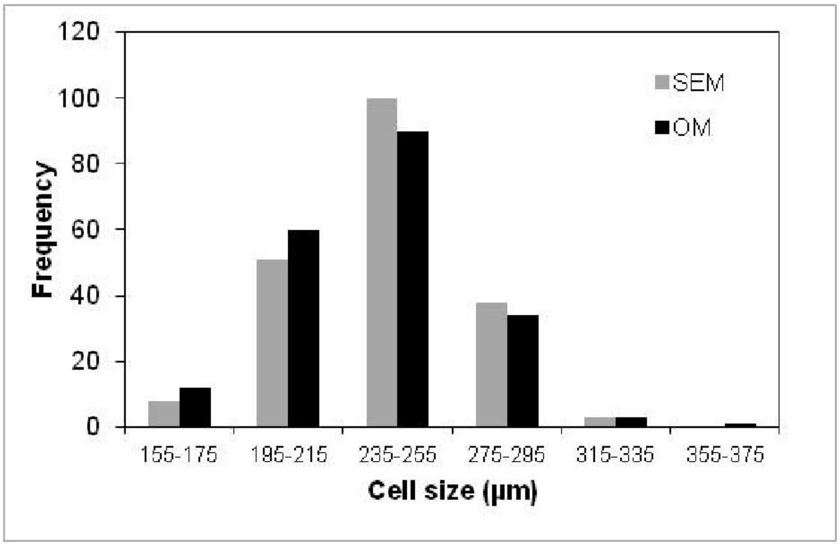

Fig.5. Comparative cell size distribution measurement obtained from SEM and the improved OM technique.

The comparable results have confirmed that foam cells images obtained with the proposed coating and can be utilized to quantitatively characterize the morphology PE foams. With the portability of the proposed optical microscopy setup, extension of its usage in other related field of study related to PE foams can be initiated and one example of this application is going to be discussed in the next section.

3.3 Application of the improved OM technique during compression test

Fig.6 shows the application of the customized optical microscopy setup in evaluating real-time compression deformation of the contrast enhanced PE foam sample. The mini portable digital microscope was located beside the compression jig of the universal testing machine and it was used to monitor progressive collapse of foam cell. The described setup shows the versatility of the proposed optical microscopy technique and the procedure is impossible to be implemented if an electron microscope was used for the intended observation technique.

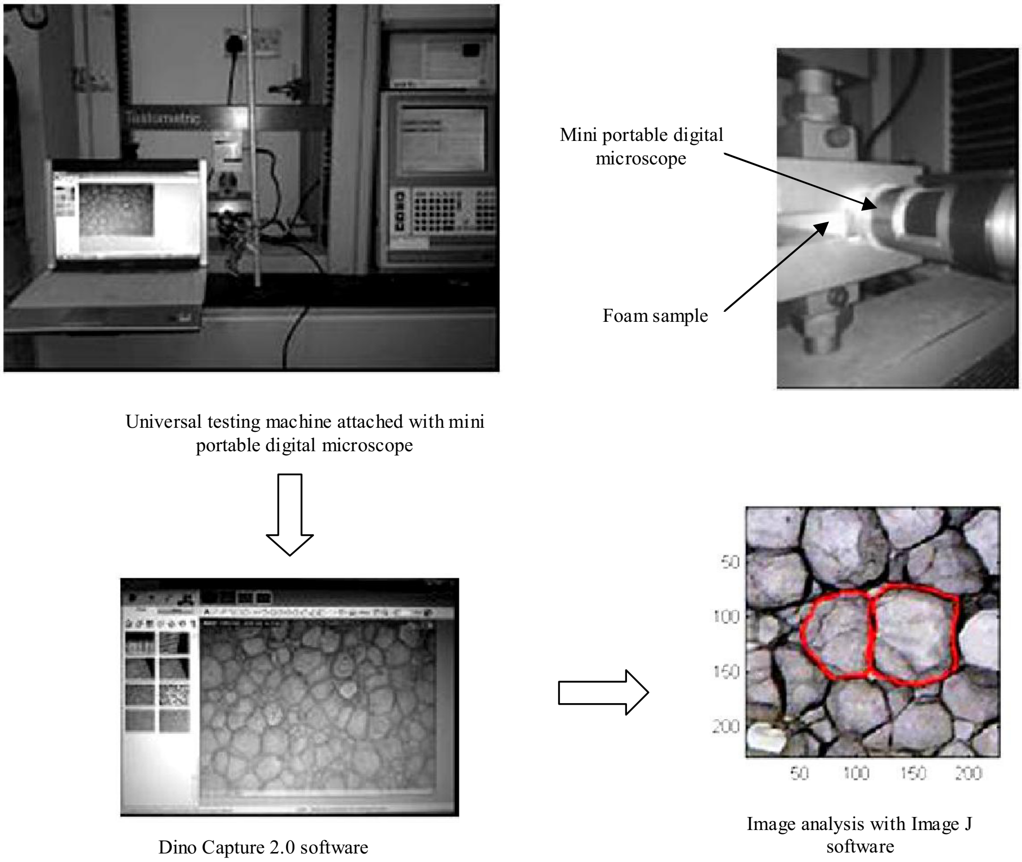

Fig.6.Compression test experimental setup and image analysis used in the improved OM technique

The success of the above setup can be evaluated through real-time images obtained during the compression test. This is shown in Fig.7, where series of enhanced-contrast images of the foam cells microstructure changes clearly display several characteristics or stages of cell deformation such as cell wall bending, buckling and ultimately cell collapse. The quality of images captured using the proposed technique seems to match similar findings obtained using more sophisticated imaging instrument such as 3D micro-tomography[9,10,11].

Conclusion

The proposed of black soot coating technique has managed to solve contrast problem available during the observation of PE foam cell via optical microscope. Coupled with polarized light, the technique can be used to eliminate glare problem resulted from light scattering and reflection which usually exists in observation white materials using optical microscopy. Samples with black soot coating give better image quality and offer higher contrast that clearly underlines the cell boundaries compared to those coated with Au/Pd coating. The success of real-time monitoring of foam cell deformation during compression test proved that the proposed simple image enhancement was practical within the intended application and more important, affordable compared to other advanced microscopy technique.

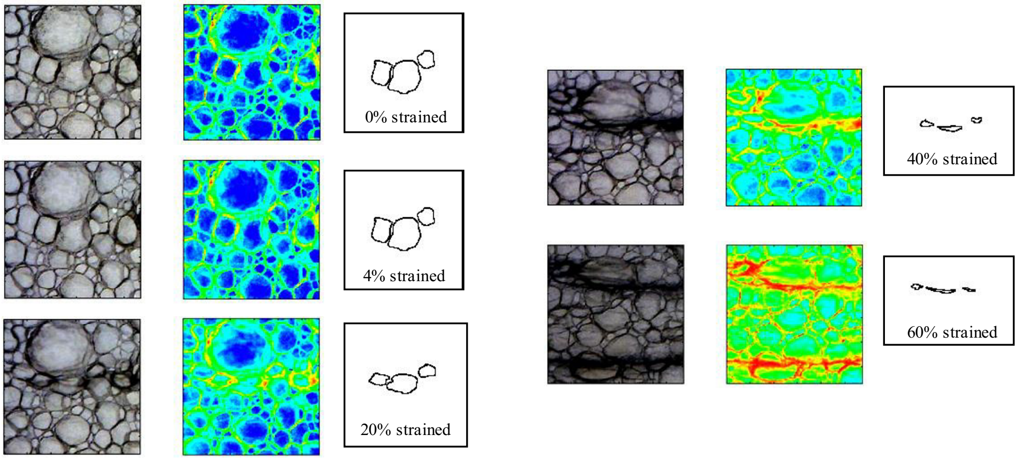

Fig. 7. Progressive deformation images of PE foam sample during compression test using the porposed OM technique captured at different compressive strains; a) initial undeformed state ;(b) 4% ;(c) 20% ;(d) 40% ;(e) 60%

Acknowledgements

The authors would like to acknowledge the financial support given by the Malaysian Ministry of Higher Education through the Fundamental Research Grant Scheme (Grant No. 203/PBAHAN/6071172)

References

1. Raskar R, Agrawal A, Wilson CA, Veeraraghavan A. Glare aware photography: 4D ray sampling for reducing glare effects of camera lenses. *ACM Transactions on Graphics (TOG)* 2008; **27**(3):56-65
2. Landers R, Venzmer J, Boinowitz T. Methods for Cell Structure Analysis of Polyurethane Foams. In *Proceedings of The Polyurethane Conference (API)* 2005; p. 453-465
3. Sims GLA, Khunniteekool C. Cell-size Measurement of Polymeric Foams. *Cellular Polymers* 1994; **13**(2):137-146.
4. Wineland, SH, Bartz, AM. An Instrument for Measuring the Cell Size of Polystyrene and Polyethylene Foams. *Journal of Cellular Plastics* 1986; **22**:122-138
5. Pires LF, Borges FS. Passoni S, Pereira AB. Soil pore characterization using free software and a portable optical microscope. *Pedosphere* 2013; ***23***(4):503-510.
6. Faith D, Horsfield CJ, Nazarov W. (2006). Characterization of pore size of trimethylol propane triacrylate (TMPTA) polymer foam by pulsed sputter coating and SEM analysis. *J Mat Sci* 2006; **41**(13):3973-3977.
7. Mukhopadhyay SM. Chapter 9: Sample preparation for microscopic and spectroscopic characterization of solid surfaces and films. In Mitra S. *Sample Preparation Techniques in Analytical Chemistry*. New Jersey: Wiley; 2003. p. 377-411.
8. Zakaria, Z. Compressive properties and simulation of low density polyenthylene foams. *PhD Thesis* Universiti Sains Malaysia, 2013
9. Maire E, Buffiere JY, Salvo L, Blandin JJ, Ludwig W, Letang JM. On the application of X-ray microtomography in the field of materials science. *Adv Eng Mat* 2001; **3**(8):539-546.
10. Daphalapurkar NP, Hanan JC, Phelps NB, Bale H, Lu H. Tomography and simulation of microstructure evolution of a closed-cell polymer foam in compression. *Mechanics Adv Mat Struc*, 2008; **15**(8):594-611.
11. Elliott, JA, Windle, AH, Hobdell, JR, Eeckhaut, G, Oldman, RJ, Ludwig, W, & Baruchel, J. In-situ deformation of an open-cell flexible polyurethane foam characterised by 3D computed microtomography. *J Mat Sci* 2002; **37**(8):1547-1555