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Optimization of conditions for production of sago starch-based foam

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Abstract

Production of sago starch-based foam involved mixing of sago starch with polyvinyl alcohol (PVA) or polyvinyl pyrrolidone (PVP) followed by preparation of electron beam irradiated sago starch/PVA and sago starch/PVP sheets and expanding them in a microwave. The results revealed that good foams with high linear expansion and closed cell structure can be produced from 25:15 of sago starch:PVA and 30:10 of sago starch:PVA blends prepared at 80 °C and electron beam irradiated at 15 kGy or 10 kGy for the cross-linking process. An increment of sago starch in the blends enhanced the linear expansion of the foams produced. Change in the blend morphology was observed when it was exposed to higher irradiation doses as electron beam irradiation induced the cross-linking in PVA and PVP, and leaching of amylose and amylopectin from the starch granules. Sago starch/PVA blend is more suitable for foam production because it produced flexible and glossy foam as compared to sago starch/PVP blend which produced very rigid foam.

Keywords: Starch; Irradiation; Biodegradable; Foam production

1. Introduction

Single-use packaging materials have been identified as suitable items to be replaced by biodegradable materials from renewable resources which will lead to ecological benefits. Its formulation is usually composed of starches such as corn, wheat and tapioca (Bhatnagar & Hanna, 1996). Other polymers have also been incorporated such as polystyrene and polypropylene which are not biodegraded (Chinnaswamy & Hanna, 1990). Starch foams with insulating properties that are similar to polystyrene foam have been industrially developed by extrusion process (Artz, Warren, & Villor, 1990; Bastioli, Bellotti, Camia, Giudice, & Rallis, 1995; Fang & Hanna, 2001). PVA was incorporated with high amylose starches and water to produce loose-fill as commercial packaging (Kondo, Sawatari, Manley, & Gray, 1994; Nwufo & Griffin, 1985). Starch/PVA blends have demonstrated excellent compatibility but are not amendable when extruded.

Sago starch was used in this study since it is renewable, available locally and cheap. Polyvinyl alcohol (PVA) was incorporated in the formulation as it is cross-linkable polymer, completely biodegradable and cheap as compared to other biodegradable polymer (Zhai, Yoshii, Kume, & Hashim, 2002). It was reported by Shogren, Lawton, Teifenbacher, and Chen (1998) that blending of starch with PVA can improve the moisture resistance of the products. The strength of the product was also enhanced. Polyvinyl pyrrolidone (PVP) was also used in comparison with PVA as it is biodegradable and cross-linkable. Electron beam irradiation process was employed in order to induce cross-linking between the polymers. Optimum cross-linking will result in foams that will resist thermal collapse and by

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varying the irradiation doses, it will produce different degree of cross-linking between the polymers. Microwave was subsequently used as a technique to expand the irradiated blends into foams. Good foams should undergo high linear expansion and have closed cell structure (Shutov, 1991).

The objectives of this study were to develop an optimum formulation and to determine the optimum conditions for sago starch-based foam preparation. The effects of blend composition, mixing temperature and electron beam irradiation dose on linear expansion and cell structure of foams were studied.

2. Materials and methods

2.1. Materials

Sago starch was donated by Nitsei Sago Industries Sdn. Bhd, Mukah, Sarawak. Polyvinyl alcohol (PVA-117, M_w 74,000) was purchased from Kuraray Poval Co. Ltd., Japan. Polyvinyl pyrrolidone (PVP K 90, M_w 360,000) was purchased from BASF, Germany. Distilled water was used for the preparation of the blends.

2.2. Preparation of foam

Prior to preparation of the starch/PVA/water and starch/PVP/water mixtures. PVA was solubilised in distilled water at 121 °C for 10 min and left to cool. PVP, however, was solubilised in distilled water at room temperature overnight. Starch was then mixed homogeneously with aqueous PVA or PVP at room temperature or at 80 °C before irradiation. Thirty millilitres of the mixture was poured into a square petri dish (10 cm × 10 cm) to form a sheet and irradiated at 10, 15, 20, 25 or 30 kGy. The irradiation conditions of the electron beam machine were as follows:

Acceleration voltage	2.0 MeV
Beam current	10.0 mA
Conveyer speed	0.94 m/min

The irradiated sheets were then expanded by heating in a microwave model TTAG-A437 (Sharp) for 5–8 min. The temperature range was 160–200 °C. In this study, the distilled water acted as a blowing agent.

2.3. Effects of PVA, PVP and sago starch concentrations, mixing temperature and irradiation dose on foam formation

The appropriate polymer concentration, mixing temperature and irradiation dose were obtained based on linear expansion ratio of the foams produced. The ratios of sago starch and PVA or PVP in 100 ml of water was varied between 10:30 and 30:10 and they were prepared at room temperature or 80 °C. The results of a preliminary study showed that the blend cannot be prepared if the total amount of PVA or PVP and starch was more than 40 g solid. It was also observed that foams cannot be prepared from sago starch, PVA and PVP when they were irradiated alone at 10, 15, 20, 25 and 30 kGy. The ratio of PVA or PVP with sago starch and the irradiation doses employed are shown in Table 1.

2.4. Linear expansion determination

The percentage linear expansion was obtained on foaming the sago starch/PVA and sago starch/PVP blends in a microwave. The unfoamed sheets were ruled with a line across using a fine oil pen. Each line was measured before and after foaming. The percentage linear expansion was calculated as follows:

% Linear expansion = (length after foaming − length before foaming) × 100 (length before foaming)

2.5. Scanning electron microscopic studies on sago starch/PVA and sago starch/PVP blends and foams

Blends were cryogenically frozen and manually fractured in liquid nitrogen for 2–3 min. The sample was mounted on aluminium stubs and viewed using an environment scanning electron microscope model Philips XL30/TMP or a JOEL 6400 (Japan). Foams were manually cut and mounted on aluminium stubs for viewing.

2.6. Light microscopic studies on sago starch/PVA blends

Irradiated sago starch/PVA sheet was cut into 1 mm × 1 mm pieces. They were put into separate vials and

Table 1
Formulation of blends to produce foams with different ratio of PVA or PVP and sago starch at room temperature and 80 °C

Blend	Ratio of PVA/PVP (g) to Sago starch (g)	Irradiation dose (kGy)
Sago starch:PVA and Sago starch:PVP	10:30	10
		15
		20
		25
		30
	15:25	10
		15
		20
		25
		30
	20:20	10
		15
		20
		25
		30
	25:15	10
		15
		20
		25
		30
	30:10	10
		15
		20
		25
		30

fix in 40% glutaradehyde fixative for 12–24 h at 4 °C. They were then washed with 0.1 M sodium cacodylate buffer for 3 times with 10 min interval between each wash. Thereafter, they were post-fixed in 1 osmium teroxide for 2 h at 4 °C. They were then washed again with 0.1 M sodium cacodylate buffer for 3 times with 10 min interval between each wash. They were dehydrated with a series of acetone (35% for 10 min, 50% for 10 min, 75% for 10 min, 95% for 10 min and 100% for 15 min) for 3 times. Samples were infiltrated with resin and acetone mixture (1:1 for 1 h, 1:3 for 2 h, 100% resin overnight and 100% resin for 2 h). They were embedded in resin by placing them into beam capsules which were later filled with resin. They were polymerized in an oven at 60 °C for 24–28 h. Thick sectioning of the samples was prepared by using a glass knife and ultramicrotome to cut 1 μm thick sections. The sectioned were then placed onto glass slides and stained with toludine blue. They were subsequently dried on a hot plate and washed with 0.1 M sodium cacodylate and the slides were examined under a light microscope. Sago starch/PVP blend cannot be prepared for light microscope viewing because the blend dissolved in glutaraldehyde.

2.7. Physical appearance of foams

The foams were visually observed. The appearance of the skin and texture of the foams were recorded.

3. Results and discussion

3.1. Effects of PVA, PVP concentration, irradiation dose and mixing temperature on linear expansion

Linear expansion of sago starch/PVA blend prepared at room temperature is presented in Table 2. It was found that the highest linear expansion of foam produced was obtained from sago starch/PVA blends irradiated at 15 kGy. This value should coincide with the point when maximum cross-linking has occurred. A major practical use of high energy radiation to modify materials has been in the cross-linking of polymers (Rosiak, 1998). When aqueous solution of polymer was subjected to ionizing irradiation, hydroxyl radicals were the main reactive species responsible for reactivity transfer from water to polymer chain (Rosiak & Ulanski, 1999). It was reported by Huglin and Zakaria (1986) that after PVA/starch/water blend was irradiated, hydroxyl radicals could initiate PVA and starch radicals. PVA radical reacted easily with other PVA molecules to form cross-linked PVA networks. The cross-linked polymer holds on the pressure that gushed out of the blend while expanding in a microwave. Cross-linking enhanced the resistance of cellular product to thermal collapse (Ghazali, Johnson, & Dahlan, 1999). Linear expansion ratio increased when irradiation dose was increased from 10 to 15 kGy. This phenomenon was due to an increase in cross-linking occurring in the blend as a result of increased irradiation. It also indicated that the amount of cross-linking was insufficient when the blend was irradiated at 10 kGy. With an irradiation dose of 15 kGy, maximum linear expansion was obtained as sufficient cross-linking has occurred in the blend. Thereafter, the linear expansion ratio was decreased as the irradiation dose was increased from 20 to 30 kGy. With an increment in the irradiation dose, more cross-linking occurred in the blend resulting in less expansion of the foam. Excessive cross-linking inhibited the mobility of the polymer chain. The reduction in expansion of the foam might also be due to degradation of sago granules as a result of higher amounts of irradiation. From the results obtained, blends containing higher amounts of sago starch exhibited higher linear expansion ratio as can be seen from the mean linear expansion ratio of 30:10 of sago starch:PVA was the highest followed by the 25:25, 20:20, 15:25 and 10:30 of sago starch:PVA of the similar blend, consecutively.

Table 3 shows the linear expansion of sago starch/PVA blends prepared at 80 °C. The results were similar to that of sago starch/PVA blends prepared at room temperature. The maximum linear expansion was obtained when the blend was irradiated at 15 kGy followed by blends irradiated at 20, 10, 25 and 30 kGy, respectively. The blend containing 30:10 of sago starch:PVA gave the highest linear expansion followed by that with 25:15, 20:10, 15:25, and 30:10 of sago starch:PVA, respectively. From the statistical analysis, however, there were no significant differences between the linear expansion of 25:15 and 20:20, and 10:30, and, 15:25 of sago starch:PVA blends. It was noticed that the maximum linear expansion value obtained with sago starch/PVA blend at 80 °C was higher than that obtained

Table 2
Linear expansion (%) of sago starch:PVA blends prepared at room temperature

Blend composition Sago starch:PVA	Dose (kGy)					Mean**
	10	15	20	25	30	
10:30	22.44 ± 5.25	42.56 ± 8.05	26.91 ± 4.14	0.00 ± 0.00	0.00 ± 0.00	18.38 ± 17.26^{e}
15:25	36.04 ± 7.37	57.34 ± 6.90	36.52 ± 3.96	18.09 ± 6.38	15.40 ± 5.29	29.60 ± 20.34^{d}
20:20	47.72 ± 4.43	65.71 ± 7.63	38.20 ± 3.59	37.07 ± 4.04	0.00 ± 0.00	39.62 ± 17.73^{c}
25:15	76.22 ± 4.87	87.30 ± 22.26	44.96 ± 7.27	23.29 ± 2.00	0.00 ± 0.00	46.30 ± 33.50^{b}
30:10	78.57 ± 10.61	94.66 ± 5.36	64.09 ± 2.57	31.42 ± 11.51	0.00 ± 0.00	53.74 ± 35.28^{a}
Mean*	52.20 ± 23.36^{b}	69.51 ± 20.89^{a}	42.08 ± 13.30^{c}	20.77 ± 13.07^{d}	3.08 ± 6.64^{e}	$R^2 = 0.86$

* Means within the same row with same superscript are not significantly different ($P < 0.05$).
** Means within the same column with same superscript are not significantly different ($P < 0.05$).

Table 3
Linear expansion ratio of sago starch:PVA blends prepared at 80 °C

Blend composition Sago starch:PVA	Dose (kGy)					Mean**
	10	15	20	25	30	
10:30	23.09 ± 3.11	42.78 ± 3.56	45.56 ± 2.59	34.62 ± 4.47	14.40 ± 2.80	32.09 ± 12.40^{c}
15:25	24.96 ± 1.26	49.56 ± 4.30	38.26 ± 1.74	35.67 ± 2.44	17.13 ± 2.15	33.11 ± 11.63^{c}
20:20	76.49 ± 2.32	105.87 ± 4.45	70.07 ± 5.83	53.94 ± 2.91	15.51 ± 1.92	64.38 ± 30.66^{b}
25:15	65.96 ± 2.66	103.84 ± 3.57	84.17 ± 6.57	57.10 ± 5.13	22.60 ± 14.50	66.73 ± 28.66^{b}
30:10	103.13 ± 0.23	86.76 ± 12.69	52.32 ± 14.49	34.15 ± 15.17	33.60 ± 10.98	76.43 ± 31.08^{a}
Mean*	58.73 ± 31.44^{c}	81.59 ± 30.04^{a}	64.96 ± 21.16^{b}	46.73 ± 11.90^{d}	20.76 ± 11.54^{e}	$R^2 = 0.81$

* Means within a same row with same superscript are not significantly different ($P < 0.05$).
** Means within a same column with same superscript are not significantly different ($P < 0.05$).

with sago starch/PVA blend at room temperature. This was due to the fact that when the blend was heated with water, the granules swell and amylose and amylopectin would leach out of the granules. This can be observed during mixing where the viscosity of the blend increased rapidly. On cooling, the amylose and later amylopectin formed network in the paste. Therefore, when the blend was expanded in a microwave, the network in starch paste together with the cross-linking that had occurred among the PVA chains, helps trap the water inside the foam cell. This resulted in an increment in the linear expansion ratio of blends mixed at 80 °C as compared to the blend mixed at room temperature.

Linear expansion of sago starch/PVP blends prepared at room temperature is shown in Table 4. It was found that the highest linear expansion was obtained when sago the starch/PVP blend was irradiated at 10 kGy. In comparison with the linear expansion of sago starch/PVA, the dose required to achieve the maximum linear expansion of sago starch/PVP was lower. This might be due to the difference in structure between PVA and PVP. The functional group of PVP has nitrogen with lone pair electron which is more reactive than the functional group, OH, in PVA. Linear expansion of 25:15 of sago starch:PVP was higher than that of 30:10, 20:20, 15:25 and 10:30 of sago starch:PVP, respectively. These were similar to the results obtained with sago starch:PVA blends mixed at room temperature and 80 °C. However, linear expansion of sago starch:PVP blends with 30:10, 20:20 and 15:25 compositions were not significantly different. The cell structure of the blends irradiated at 25 and 30 kGy were collapsed and shrunken. This might be due to excessive cross-linking in the blends. Irradiation at 10 kGy was sufficient for preparation of sago starch/PVP foam. However, the maximum linear expansion value obtained was lower than that of sago starch/PVA prepared at 80 °C.

Linear expansion of sago starch/PVP prepared at 80 °C is presented in Table 5. From the results obtained, the highest linear expansion was attained when the blend was irradiated at 10 kGy followed by blends irradiated at 15 and

Table 4
Linear expansion (%) of sago starch:PVP blends prepared at room temperature

Blend composition Sago starch:PVA	Dose (kGy)					Mean**
	10	15	20	25	30	
10:30	33.60 ± 10.98	25.70 ± 16.99	18.06 ± 12.46	0.00 ± 0.00	0.00 ± 0.00	15.47 ± 16.95^{c}
15:25	66.94 ± 4.64	54.74 ± 3.28	39.81 ± 3.63	0.00 ± 0.00	0.00 ± 0.00	32.30 ± 28.35^{b}
20:20	73.16 ± 8.71	46.75 ± 2.62	31.14 ± 7.43	0.00 ± 0.00	0.00 ± 0.00	30.21 ± 28.98^{b}
25:15	82.73 ± 8.46	63.92 ± 8.09	61.45 ± 11.11	0.00 ± 0.00	0.00 ± 0.00	41.62 ± 35.99^{a}
30:10	77.91 ± 4.61	49.54 ± 11.09	27.95 ± 3.50	0.00 ± 0.00	0.00 ± 0.00	31.08 ± 30.87^{b}
Mean*	66.87 ± 19.18^{a}	48.13 ± 15.84^{b}	35.68 ± 16.84^{c}	0.00 ± 0.00^{d}	0.00 ± 0.00^{d}	$R^2 = 0.88$

* Means within the same row with same superscript are not significantly different ($P < 0.05$).
** Means within the same column with same superscript are not significantly different ($P < 0.05$).

Table 5
Linear expansion (%) of sago starch:PVP blends prepared at 80 °C

Blend composition Sago starch:PVP	Dose (kGy)					Mean**
	10	15	20	25	30	
10:30	72.51 ± 8.62	56.28 ± 4.67	34.70 ± 11.30	4.02 ± 9.85	0.00 ± 0.00	33.50 ± 29.83^{c}
15:25	94.40 ± 2.80	72.87 ± 10.79	44.96 ± 12.11	0.00 ± 0.00	0.00 ± 0.00	42.44 ± 39.28^{b}
20:20	81.62 ± 40.57	75.85 ± 10.48	94.75 ± 19.83	16.20 ± 39.68	0.00 ± 0.00	53.68 ± 46.27^{a}
25:15	103.81 ± 5.67	71.07 ± 3.28	40.91 ± 3.13	0.00 ± 0.00	0.00 ± 0.00	43.16 ± 41.27^{b}
30:10	94.40 ± 4.85	67.78 ± 4.79	26.39 ± 16.39	0.00 ± 0.00	0.00 ± 0.00	37.71 ± 39.00^{cb}
Mean*	89.35 ± 20.79^{a}	68.77 ± 9.79^{b}	48.34 ± 27.57^{c}	4.04 ± 18.13^{d}	0.00 ± 0.00^{bd}	$R^2 = 0.82$

* Means within the same row with same superscript are not significantly different ($P < 0.05$).
** Means within the same column with same superscript are not significantly different ($P < 0.05$).

20 kGy. When the blends were irradiated at 25 kGy, there were thermal collapses for 15:25 and 25:15 sago starch:PVP. All of the blends were thermal collapsed when they were irradiated at 30 kGy. This was similar to that obtained when sago starch/PVP blends were prepared at room temperature and irradiated at 25 and 30 kGy. As for the blend composition, the 20:20 of sago starch:PVP blend exhibited the highest linear expansion ratio as compared to

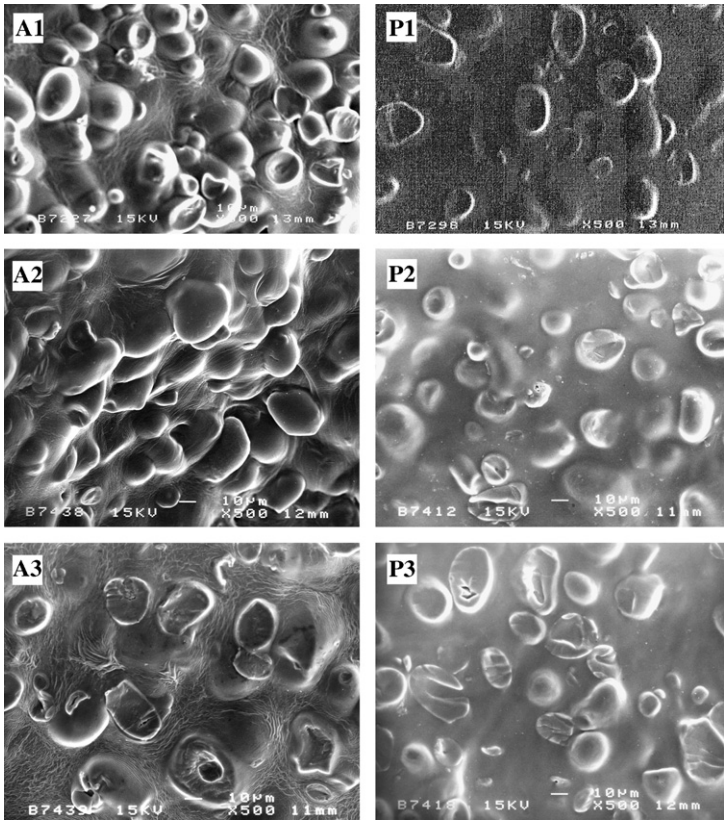

Fig. 1. SEM micrographs of 20:20 of sago starch:PVA (A) and 20:20 of sago starch:PVP (P) blends irradiated at 10 kGy (A1, P1), 20 kGy (A2, P2) and 30 kGy (A3,P3).

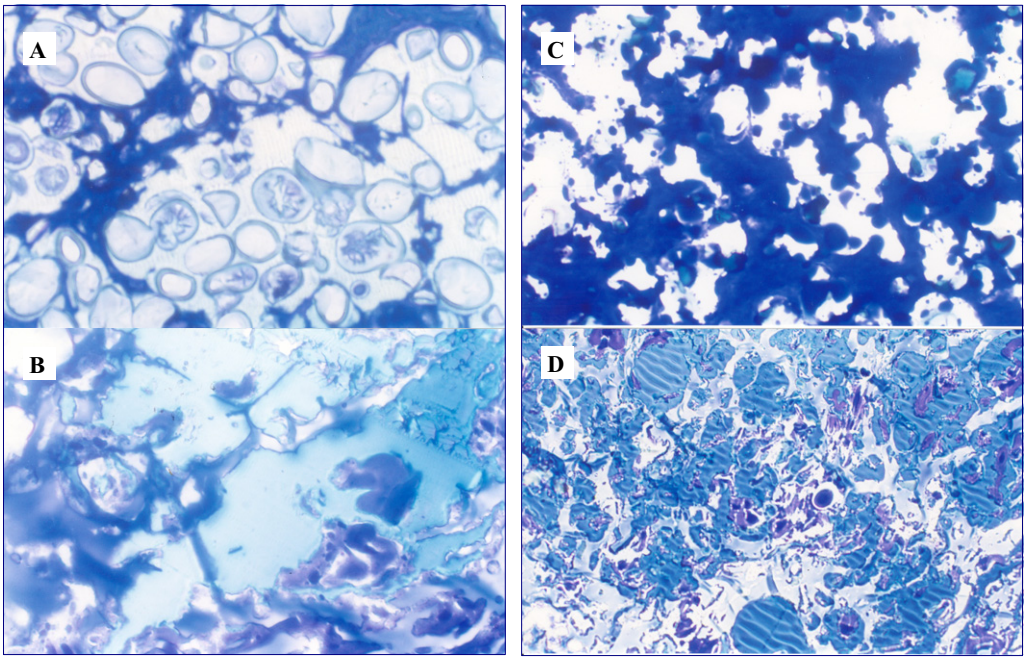

Fig. 2. Light photomicrographs of sago starch:PVA blends.

the 25:15, 30:10 and 10:30 of sago starch:PVP blends. However, the linear expansion of sago starch/PVP blend prepared at 80 °C was higher than that of sago starch/PVP blend prepared at room temperature. It is noticeable that at 10 kGy irradiation dose, sago starch/PVP blend had higher linear expansion ratio than sago starch/PVA foam. This might be due to the higher gel content obtained when sago starch/PVP blend was irradiated at 10 kGy.

From the overall statistical analysis, it was found that mixing the blends at 80 °C for sago starch/PVA or sago starch/PVP produced foams with significantly higher linear expansion when compared to that prepared at room temperature. The average linear expansion values for sago starch/PVA and sago starch/PVP mixed at room temperature and 80 °C were 33.83% and 48.33%, respectively. The results also show that there were significantly different linear expansion values obtained for sago starch/PVA and sago starch/PVP blends. The sago starch/PVA blend had higher expansion than sago starch/PVP blend which were averaged as 46.04% and 36.12%, respectively.

Blend composition play an important role in foam preparation. From the results, it was found that starch:PVA/PVP compositions, 30:10 and 25:15, produced foams with high linear expansion with averaged values of 49.45% and 19.75%, respectively. They are significantly different. 20:20 of sago starch:PVA/PVP produced foams with significantly lower linear expansion than 30:10 of sago starch:PVA/PVP and 25:15 of sago starch:PVA/PVP. The linear expansion value was 46.97%. Sago starch:PVA/PVP with 15:25 and 10:30 ratios produced foams with the lowest linear expansion. The values were 34.36% and 24.86%, respectively.

Irradiation dose also played a major role in foam preparation. The results of statistical analysis confirmed the role of irradiation on linear expansion ratio where 10 kGy and 15 kGy were the optimum irradiation doses for good expansion of foams produced from sago starch/PVP and sago starch/PVA, respectively.

3.2. Scanning electron microscopic studies of blends

Microscopy can be used as a tool to obtain information on domain size and size distribution in the blends. Blend morphology of 20:20 of sago starch:PVA and 20:20 of sago starch:PVP is shown in Fig. 1. This ratio was selected for the study because the results of a preliminary study showed that clearer results could be obtained as compared to that

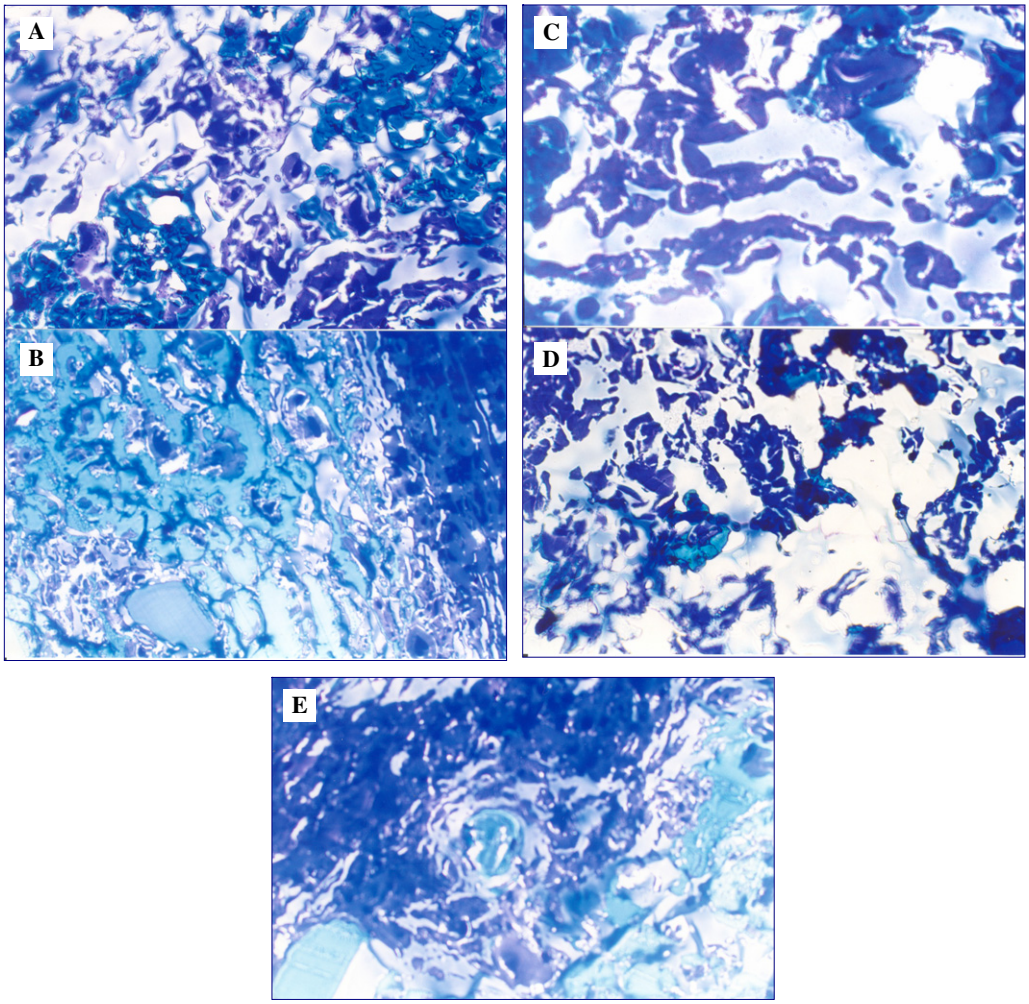

Fig. 3. Light photomicrographs of sago starch:polyvinyl alcohol blends mixed at 80 °C and irradiated at (A)10, (B) 15, (C) 20, (D) 25 and (E) 30 kGy.

of 25:15 and 30:10 of sago starch:PVA blends due to less starch being scattered in the blend among the PVA. It is interesting to note that irradiation changed the phase morphology of the blends. The scanning electron micrographs revealed that at 10 kGy irradiation, granules were found embedded in the matrix whereas at 20 and 30 kGy, the granules were on the surface. The fractured surface of sago starch/PVA blend irradiated at 30 kGy appeared rougher and more brittle than that irradiated at 10 and 20 kGy. Fig. 1 (A3) had more cracks appearing on the surface. The development of these fractures was attributed to the presence of excessive cross-linking. The surfaces of the blends irradiated at 10 and 20 kGy were more regular. There were no cracks observed, implying a lower degree of cross-linking that resists crack propagation. This was reflected by the lower strength of the blend. This was due to the fact that there was more cross-linking formed when the blends were exposed to higher irradiation dose as compared to that exposed to a lower dose (Ratnam, Nasir, Baharin, & Zaman, 2001). Therefore, the SEM studies further confirmed the occurrence of irradiation-induced cross-linking in the blend. When the irradiation dose was increased, the elasticity of sago starch/PVA blend was increased. Scanning electron micrographs of sago starch/PVP blend are shown in Fig. 1 (P1, P2 and P3). Blends of sago starch/PVP were sticky and elastic when they were exposed to 10 and 15 kGy irradiation. When the irradiation dose was increased from 20 to 30 kGy, the texture of sago starch/PVP blend was soft, brittle and less sticky.

3.3. Light microscopic studies on sago starch/PVA blend

Light microscopy provides a general characterization of the granular morphology (Andreas, Hansen, Schulz, Jørgensen, & Donald, 2003). Fig. 2 shows the effect of temperature on the leaching of amylose and amylopectin in the 10:30 of starch:PVA and 30:10 of sago starch:PVA blends. The blue area shows the leaching of amylose and amylopectin from the starch granules. It can be seen that when the blend was heated at 80 °C, the granule shape was invisible. Blue colour was observed all over the micrograph because more amylose and amylopectin have leached out of the granules. During heating at the above gelatinization temperature, the starch granules absorb water, swell and undergo gelatinization. The viscosity of the starch suspension increased. The starch granules

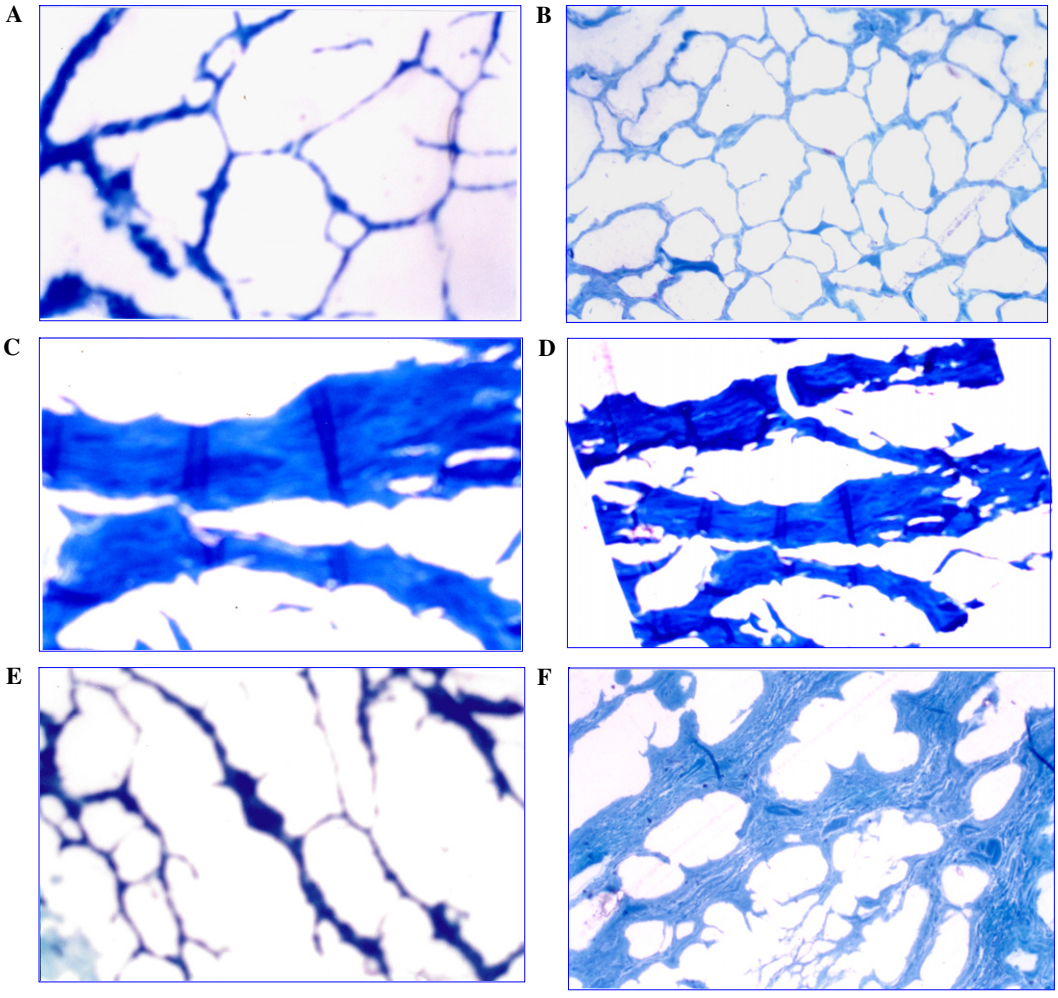

Fig. 4. Light photomicrographs of sago starch gel.

become distorted, and the soluble fraction of the starch (amylose and amylopectin) is released (Bilidaeris & Zawistowski, 1990). These results further explained why higher linear expansion of foam was obtained when the blend mixing temperature was 80 °C. From the results obtained earlier, the 30:10 of sago starch:PVA has higher linear expansion ratio than 10:30 of sago starch:PVA. As it can be observed from the light micrographs, the leaching of sago starch in 30:10 of sago starch:PVA was more than in 10:30 of sago starch:PVA due to higher amount of sago starch present in the former. Fig. 3 shows the effect of irradiation dose on leaching of amylose and amylopectin. The leaching was increased when the irradiation dose was increased. This was because the electron beam chopped the amylose and amylopectin into shorter chains which can diffuse out of the granules more easily. Cross-linking achieved at 15 kGy in the sago starch/PVA blend was optimum for obtaining foam with highest linear expansion. Higher amounts of cross-linking which occurred when the blends were irradiated at 20, 25 and 30 kGy restricted foam expansion although there was more leaching of amylose and amylopectin. Fig. 4 confirmed that when the irradiation dose was increased, the leaching of amylose and amylopectin was also increased.

3.4. Scanning electron microscopic studies of foams

Fig. 5 shows morphology of foams produced using 20:20 of sago starch:PVA blends that have been irradiated at 10–30 kGy. At an irradiation dose of 10 kGy, opened-cell foam was produced. This was due the fact that cross-linking in the blend was not sufficient thus the walls of the foam cells were not strong enough to hold on the vapour that gushed out of the foams. This further confirmed the linear expansion results. The biggest foam cell size was obtained when the blend was irradiated at 15 kGy. This showed that optimum cross-linking in the blends was attained. However, the cell size decreased when the irradi-

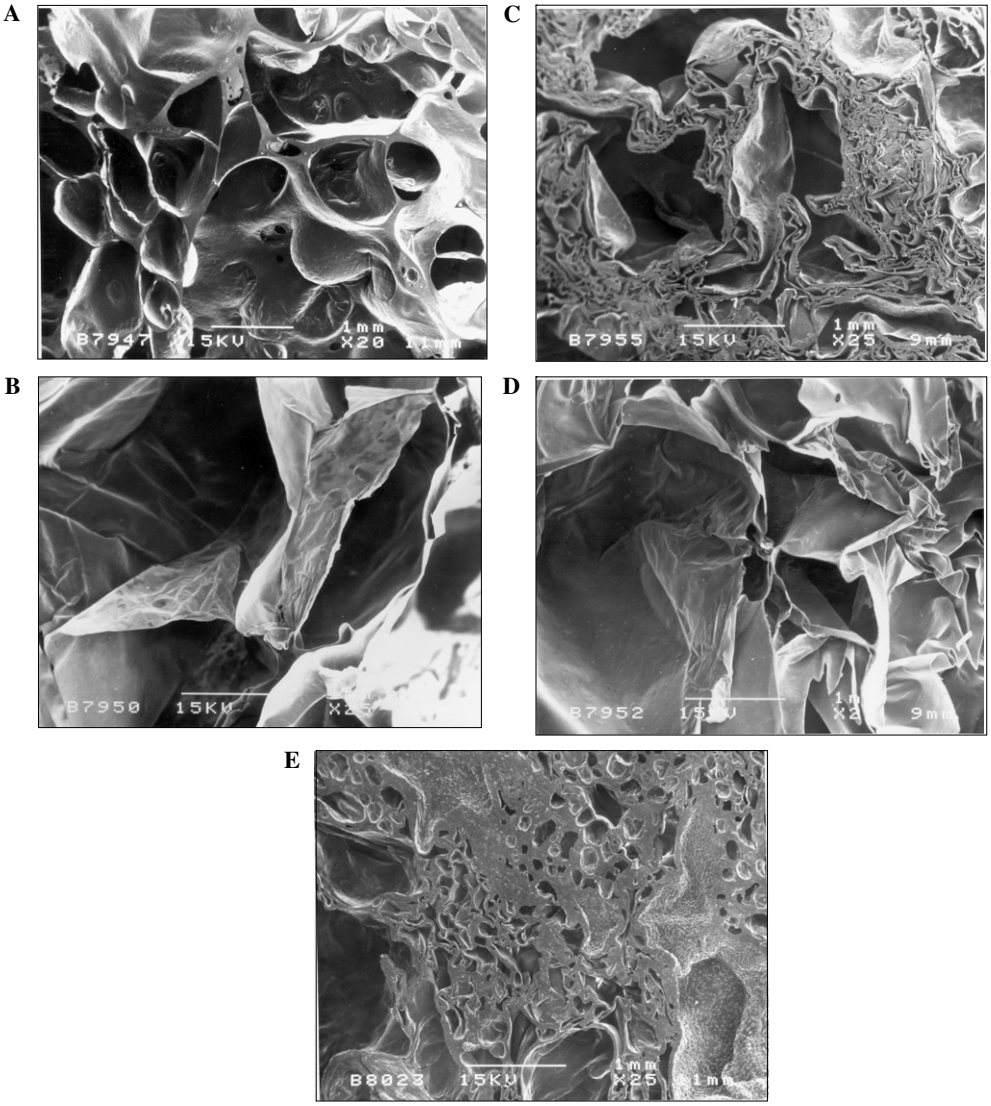

Fig. 5. Scanning electron micrographs of 20:20 of sago starch:PVA foams prepared from blends mixed at 80 °C and irradiated at (A) 10, (B) 15, (C) 20, (D) 25 and (E) 30 kGy.

ation dose was increased from 15 to 20 kGy. Further increment to 25 kGy and 30 kGy resulted in thermal collapse. This might be due to excessive cross-linking which had occurred in the blends. The findings suggested that there was a need to control the degree of cross-linking in the blends in order to achieve the desired properties of end products. The correct amount of cross-linking by irradiation is essential so as to not over or under cross-link the polymer. In comparison with closed cell foamed polymers, opened cell foams have a higher absorption capacity for water and vapour, less effective insulation capabilities for either heat or electricity, and a better ability to absorb damp (Shutov, 1991). An electron beam irradiation dose of 15 kGy was found to be suitable in the production of sago starch/PVA foams because the biggest foam cell size was obtained. Moreover, the foam cells were of closed cell type which has lower water absorption capacity than opened cell foam.

Fig. 6 shows the morphology of foams produced using 20:20 of sago starch:PVP blends that have been irradiated at 10–30 kGy. It can be seen that cell size of the foam was the biggest when the blend was irradiated at 10 kGy. This shows that sufficient cross-linking have occurred in the blend. When the irradiation dose was further increased, the cell size decreased. At 30 kGy irradiation dose, the cell was shrunken. This was due to excessive cross-linking has occurred in the blend.

Uniformity of cell size of sago starch/PVA blend irradiated at 15 kGy and sago starch/PVP blend irradiated at 10 kGy suggests that gas pressure was evenly distributed and the cell walls were cooled at the same rate. Guan and Hanna (2003) reported that foam had higher mechanical properties when cells were uniform in size, evenly distributed, hexagonal/pentagonal in shape, and unbroken, large cell and thin cell walls often resulted in lower density.

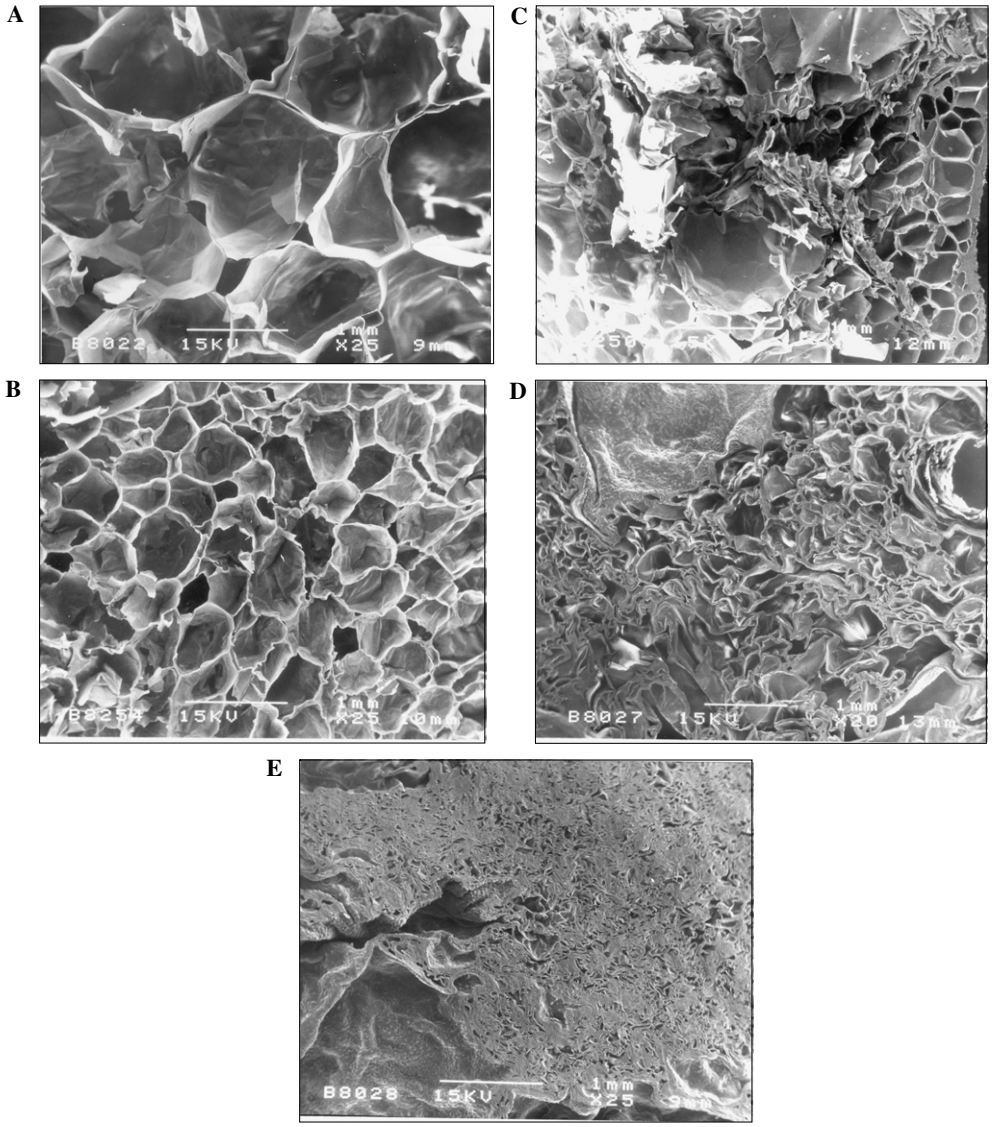

Fig. 6. Scanning electron micrographs of 20:20 sago starch:PVP foams prepared from blends mixed at 80 °C and irradiated at (A) 10, (B) 15, (C) 20, (D) 25 and (E) 30 kGy.

3.5. Physical characteristics of sago starch/PVA and sago starch/PVP foams

Sago starch/PVA foams prepared at 80 °C were smoother, more glossy, more flexible and had higher linear expansion than that prepared at room temperature. The size of foams produced from 30:10 of sago starch/PVA blend was bigger than that from 10:30 of sago starch/PVA blend when they were mixed at 80 °C and irradiated at 15 kGy. From the overall observation, sago starch/PVP foam was rigid and not glossy as compared to sago starch/PVA foam.

4. Conclusions

Good foams with high linear expansion and closed cell structure can be produced from 25:15 of sago starch:PVA and 30:10 of sago starch:PVA blends prepared at 80 °C and irradiated at 15 kGy or 10 kGy for the cross-linking process. An increment of sago starch in the blends enhanced the linear expansion of the foams produced. Change in the blend morphology was observed when it was exposed to higher irradiation doses as electron beam irradiation induced the leaching of amylose and amylopectin from the starch granules. Sago starch/PVA blend is more suitable for foam production because it produced flexible and glossy foam as compared to sago starch/PVP blend which produced very rigid and less glossy foam.

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References

Andreas, B., Hansen, M., Schulz, A., Jørgensen, K., & Donald, A. M. (2003). The molecular deposition of transgenically modified starch in the starch granules as imaged by functional microscopy. *Journal of Structural Biology, 143*, 229–241.

Artz, W. E., Warren, C., & Villor, R. (1990). Twin-screw extrusion modification of a corn fiber and corn starch extruded blend. *Journal of Food Science, 55*(3), 746–754.

Bastioli, C., Bellotti, V., Camia, M., Giudice, L. D., & Rallis, A. (1995). Starch/vinyl–alcohol copolymer interactions. In Y. Doi & K. Fukada (Eds.), *Biodegradable plastics and polymers* (pp. 200–213). Amsterdam, Netherlands: Elsevier Science B.V.

Bhatnagar, S., & Hanna, M. A. (1996). Starch-based plastic foams from various starch sources. *Cereal Chemistry, 73*(5), 601–604.

Bilidaeris, C. G., & Zawistowski, J. (1990). Viscoelastic behavior of aging starch gels: Effects of concentration, temperature, and starch hydrolysates on network properties. *Cereal Chemistry, 67*, 240–246.

Chinnaswamy, R., & Hanna, M. A. (1990). Extruded snacks food: relationship between ingredient chemical quality and expansion properties. *International Food Ingredients, 3*, 3–9.

Fang, Q., & Hanna, M. A. (2001). Characteristics of biodegradable Mater-Bi®-starch based foams as affected by ingredient formulations. *Industrial Crops and Products, 13*, 219–227.

Ghazali, Z., Johnson, A. F., & Dahlan, K. Z. (1999). Radiation crosslinked thermoplastics natural rubber (TPNR) foams. *Radiation Physics and Chemistry, 55*, 73–79.

Guan, J., & Hanna, M. A. (2003). Functional properties of extruded foam composites of starch acetate and corn cob fiber. *Industrial Crops and Products, 19*(3), 255–269.

Huglin, M. B., & Zakaria, M. B. (1986). Swell properties of copolymeric hydrogels prepared by gamma irradiation. *Journal of Applied Polymer Science, 31*, 457–475.

Kondo, T., Sawatari, C., Manley, R. S. J., & Gray, D. G. (1994). Characterization of hydrogen bonding in cellulose-synthetic polymer blend systems with regioselectivity substituted methylcellulose. *Macromolecules, 27*, 210–215.

Nwufo, B. T., & Griffin, G. J. L. (1985). Microscopic appearance of fracture surfaces and mechanical properties of starch-extended poly(vinyl alcohol). *Journal of Polymer Science: Polymer Chemistry, 23*(7), 2023.

Ratnam, C. T., Nasir, M., Baharin, A., & Zaman, K. (2001). Effect of electron beam irradiation on poly(vinyl chloride)/epoxidized natural rubber blend in presence of Trimethylopropane Triacrylate. *Journal of Applied Polymer Science, 81*, 1926–1935.

Rosiak, J. M. (1998). Gel/sol analysis of irradiated polymers. *Radiation Physics Chemistry, 51*(1), 13–17.

Rosiak, J. M., & Ulanski, P. (1999). Syntheses of hydrogels by irradiation of polymer in aqueous solution. *Radiation Physics and Chemistry, 55*, 139–151.

Shogren, Lawton, R. L., Teifenbacher, J. W. L., & Chen, K. F. (1998). Starch-poly (vinyl alcohol) foamed articles prepared by a baking process. *Journal Applied Polymer Science, 68*, 2129–2140.

Shutov, F. A. (1991). Chapter 3: Cellular structure and properties of foamed polymers. In *Handbook of polymeric foams and foam technology* (pp. 7–45). USA: Hanser pulishers.

Zhai, M., Yoshii, F., Kume, T., & Hashim, K. (2002). Syntheses of PVA/starch grafted hydrogels by irradiation. *Carbohydrate Polymers, 50*, 295–303.