

## Copolymerization of Methacrylic Acid (MAA) and Butyl Acrylate (BuA) via Emulsion Polymerization Technique

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Copolymerization of methacrylic acid (MAA) and butyl acrylate (BuA) monomers to produce copolymer nanoparticles of poly(methacrylic acid-co-butyl acrylate), p(MAA-co-BuA) was successfully synthesized via emulsion polymerization technique. Homopolymers of poly(methacrylic acid), p(MAA) and poly(butyl acrylate), p(BuA) were also successfully prepared via a similar method, which acted as control for comparison. The emulsion polymerization composed of MAA and BuA monomers, sodium dodecyl sulphate (SDS) (surfactant), potassium persulfate (KPS) (initiator) and water as dispersion medium. The effects of different molar ratios of MAA and BuA towards the formation, morphology, thermal stability and solubility of copolymer nanoparticles were thoroughly studied. The formation of copolymer nanoparticles was confirmed by the absorption peaks from Fourier transform infrared spectroscopy (FTIR). The particle size and morphology of the copolymer nanoparticles were obtained by using scanning electron microscope (SEM). Irregular shapes of the polymer nanoparticles were observed in the range of 200 – 900 nm. Thermogravimetric analysis (TGA) was utilised to compare the thermal degradation of the copolymer nanoparticles of different monomer ratios. Solubility test of the copolymer nanoparticles was performed and p(MAA-co-BuA) of the ratio of 4:1 was found to be the optimum ratio that showed improved hydrophilicity among the copolymer nanoparticles.

**Key words:** Hydrophilic polymer, copolymer, methacrylic acid, butyl acrylate, emulsion polymerization

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Nanoparticles refer to particles that have the diameter size in the range of 1-100 nanometer that are used in research studies in nanotechnology. Amongst numerous types of nanoparticles, polymeric nanoparticles (PNPs) are widely used in various fields such as electronics [1], sensor [2], medicine [3 – 4] and pollution control [5]. The polymeric nano-revolution in medicine is one of the most common strategies that has been practised in research as it provides advantages in nanomedicine, especially in smart drug delivery system [6]. NPs pose a great potential as drug carriers that act as a formulation to carry medicinal compound to a target organ in the body to achieve its curative effect effectively [7].

There are various methods to synthesize polymeric nanoparticles, where they can be prepared directly from preformed polymers or by direct polymerizations of monomers using polymerization methods. The most common polymerization used are emulsion, miniemulsion, microemulsion, interfacial and control/radical polymerization [8]. Emulsion polymerization is an emulsification process that

involves main “ingredients” such as water soluble initiator, water insoluble monomers, water and surface active agents or surfactants.

Synthetic copolymers are nowadays one of the important research that being carried out by many researchers. One of the examples is the biodegradable tri-block copolymer poly(lactic acid)-poly(ethylene glycol)-poly(L-lysine)(PLA-PEG-PLL) [9]. Copolymerization is another alternative approach to improve the exaggerated poorer qualities in certain homopolymers. Copolymerization is widely used to synthesize chain polymers consisting more than one monomer. It is proven that many copolymerization products show better qualities of the parent homopolymers [10]. One pot synthesis is one of the common strategies in copolymerization to enhance the efficiency of a chemical reaction. A chemical reaction that involves multiple steps can be easily carried out just in a single flask.

Hence, in this study, the homopolymerization and copolymerization of methacrylic acid (MAA) and

butyl acrylate (BuA) monomers were thoroughly discussed. The copolymerization of MAA and BuA is expected to improve the biodegradability and solubility of the copolymers, which may have the potentiality to be applied in any necessary fields, especially in biomedicine and medical applications. One pot emulsion polymerization was applied to synthesize homopolymers and copolymers of MAA and BuA, which composed of water as dispersion medium, sodium dodecyl sulphate (SDS) as anionic surfactant and potassium persulfate (KPS) as water soluble initiator. MAA and BuA were selected in this study due to their biodegradability.

## MATERIAL AND METHOD

### Synthesis of poly(methacrylic acid) (p(MAA)) and poly(butyl acrylate) (p(BuA)) homopolymer nanoparticles via emulsion polymerization

The emulsion polymerization reaction was conducted in 250 ml three-neck round bottom flasks equipped with magnetic stirrers and thermometers, attached to a reflux-condenser and connected with nitrogen gas inlet. The reaction was composed of methacrylic acid (MAA) monomers, sodium dodecyl sulphate (SDS) and distilled water. Nitrogen gas was bubbled into the mixture for 15–20 min and the mixture was stirred at 400 rpm/min. The flask containing the mixture solution was immersed in an oil bath of the temperature of 90 °C and aqueous potassium persulfate (KPS) solution was added into the mixture as the initiator. The polymerization was performed for 3 h. Table 1 shows the formulation of the emulsion polymerization of the p(MAA) and p(BuA) nanoparticles.

**Table 1.** Formulation of emulsion polymerization of p(MAA) and p(BuA) nanoparticles.

	p(MAA) nanoparticles (g)	p(BuA) nanoparticles (g)
Monomer	0.64	1.37
SDS	0.19	0.027
KPS	0.10	0.009
Distilled Water	74	74

**Table 2.** Formulation of copolymerization of p(MAA-co-BuA) nanoparticles at different molar ratios via emulsion polymerization.

Molar Ratio of MAA : BuA	Mass of MAA (g)	Mass of BuA (g)
1:1	0.64	0.93
1:2	0.64	1.85
1:3	0.64	2.78
1:4	0.64	3.70
Molar Ratio of BuA : MAA	Mass of BuA (g)	Mass of MAA (g)
1:2	0.93	1.27
1:3	0.93	1.91
1:4	0.93	2.55

### Copolymerization of poly(methacrylic acid-co-butyl acrylate) (p(MAA-co-BuA)) nanoparticles via emulsion polymerization

Poly(methacrylic acid-co-butyl acrylate) (p(MAA-co-BuA)) nanoparticles were prepared by one pot synthesis of emulsion copolymerization which composed of a series of different molar ratios of methacrylic acid (MAA) and butyl acrylate (BuA) monomers (Table 2). The emulsion polymerization was carried out under similar conditions to the homopolymerization of p(MAA) and p(BuA) nanoparticles.

### Characterization of Polymer Nanoparticles

The polymer nanoparticles were dried using turbovap or freeze dryer machine. The characterization of the polymer nanoparticles was performed by using Fourier transform infrared spectroscopy (FTIR), thermogravimetric analyzer (TGA), scanning electron microscope (SEM) and simple solubility test.

### Fourier Transform Infrared (FTIR)

The FTIR of p(MAA), p(BuA) and p(MAA-co-BuA) nanoparticles in solid state was recorded by using Perkin Elmer of Spectrum 100. KBr was used to prepare the solid sample for FTIR. A background spectrum with no sample in the beam was measured, followed by the measurement of spectrum with p(MAA), p(BuA) and p(MAA-co-BuA) samples, covering the scan range of 400-4000  $\text{cm}^{-1}$  [11].

### Thermogravimetric Analyzer (TGA)

TGA is a technique in which upon heating a material, its weight increases or decreases. Small amounts of p(MAA), p(BuA) and p(MAA-co-BuA) samples were placed in a TGA pan. The samples were heated at the rate of 10°C/min. Nitrogen gas was purged into the TGA to isolate the effects of degradation and avoid any misleading oxidation reactions that can possibly affect the data [12]. The samples were observed using Perkin Elmer instrument Pyris 6 TGA.

### Scanning Electron Microscopy (SEM)

Polymer samples were cut to the size small enough to fit in the sample chamber. Next, the samples were coated with gold using JFC-1600 Auto Fine Coater to improve image resolution. The samples were observed using Oxford Instrument attached to JSM-6360 LA Analytical Scanning Microscope at magnification observation of 5000×, 50 nm scale and acceleration voltage of 15 kV.

### Solubility Test

A sample of each of the polymer nanoparticles (5 mg) was dissolved in distilled water (6 mL). The time for the polymer samples to fully dissolve in the aqueous medium was recorded.

## RESULTS AND DISCUSSION

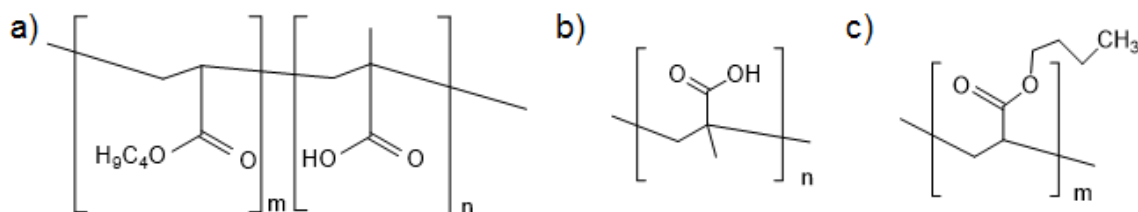
### Synthesis of Homopolymer and Copolymer Nanoparticles via Emulsion Polymerization Technique

A series of poly(methacrylic acid-co-butyl acrylate) (p(MAA-co-BuA)) copolymer nanoparticles were successfully synthesized via emulsion polymerization method at different molar ratios of MAA:BuA (1:4) and BuA:MAA (1:4) monomers (Table 3). Homopolymers of poly(methacrylic acid) (p(MAA)) and poly(butyl acrylate) (p(BuA)) nanoparticles which acted as control in this study were also successfully prepared by similar polymerization technique.

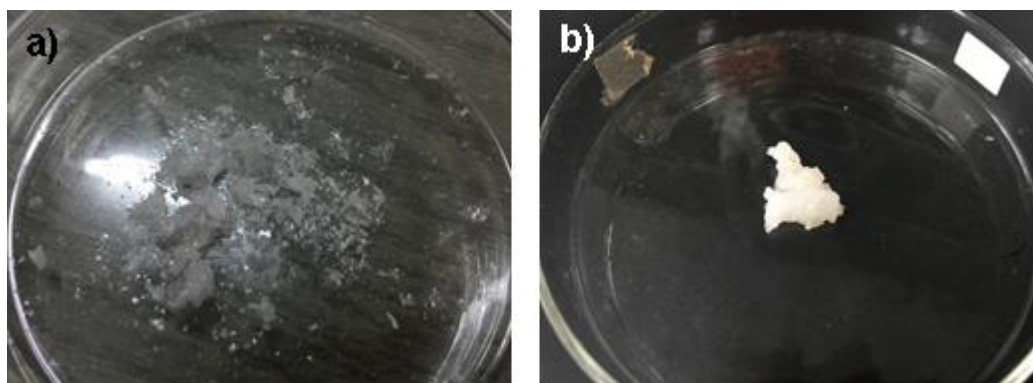
The emulsion polymerization was stabilized with the aid of an anionic surfactant, sodium dodecyl sulphate (SDS), initiated using potassium persulfate (KPS) solution in water, as medium of polymerization. The polymerization took place at 90°C for 3 hours, which resulted in the formation of insoluble precipitate, after solvent removal. Scheme 1 shows the molecular structures of p(MAA-co-BuA), p(MAA) and p(BuA) nanoparticles.

**Table 3.** Different molar ratios of p(MAA-co-BuA) nanoparticles.

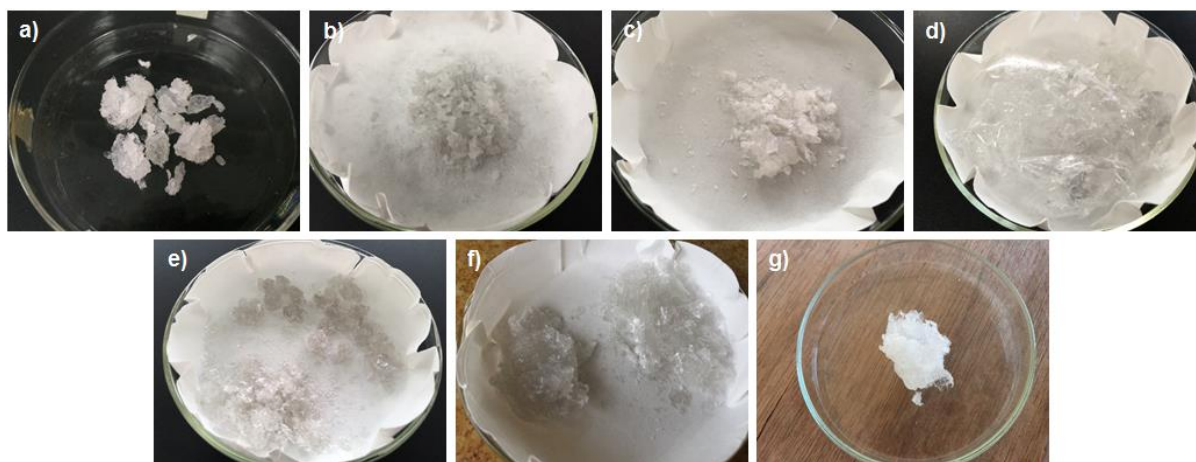
Molar Ratios of p(MAA-co-BuA) nanoparticles		
Polymer Nanoparticles	Methacrylic acid (MAA)	Butyl Acrylate (BuA)
p(MAA-co-BuA <sub>1:1</sub> )	1 (0.1M)	1 (0.1M)
p(MAA-co-BuA <sub>1:2</sub> )	1 (0.1M)	2 (0.2M)
p(MAA-co-BuA <sub>1:3</sub> )	1 (0.1M)	3 (0.3M)
p(MAA-co-BuA <sub>1:4</sub> )	1 (0.1M)	4 (0.4M)
p(MAA-co-BuA <sub>2:1</sub> )	2 (0.2M)	1 (0.1M)
p(MAA-co-BuA <sub>3:1</sub> )	3 (0.3M)	1 (0.1M)
p(MAA-co-BuA <sub>4:1</sub> )	4 (0.4M)	1 (0.1M)



**Scheme 1.** The molecular structures of (a) p(MAA-co-BuA), (b) p(MAA) and (c) p(BuA) nanoparticles.



**Figure 1.** Physical appearances of (a) p(MAA) and (b) p(BuA) nanoparticles.



**Figure 2.** Physical appearances of (a) p(MAA-co-BuA<sub>1:1</sub>), (b) p(MAA-co-BuA<sub>1:2</sub>), (c) p(MAA-co-BuA<sub>1:3</sub>), (d) p(MAA-co-BuA<sub>1:4</sub>), (e) p(MAA-co-BuA<sub>2:1</sub>), (f) p(MAA-co-BuA<sub>3:1</sub>) and (g) p(MAA-co-BuA<sub>4:1</sub>).

### Physical Appearance of p(MAA) and p(BuA) Homopolymer Nanoparticles and p(MAA-co-BuA) Copolymer Nanoparticles

The synthesis of p(MAA) and p(BuA) nanoparticles was conducted by using formulations as shown in Figure 1. Both p(MAA) and p(BuA) nanoparticles revealed different behaviours during emulsion polymerization, where the emulsion dispersion of p(MAA) nanoparticles remained as colourless solution throughout the polymerization. In contrast, emulsion dispersion of p(BuA) nanoparticles turned cloudy after three hours of polymerization. The end product of p(MAA) nanoparticles appeared as a clear solid crystal-like form (Figure 1(a)), while p(BuA) nanoparticles appeared as a sticky white solid form (Figure 1(b)).

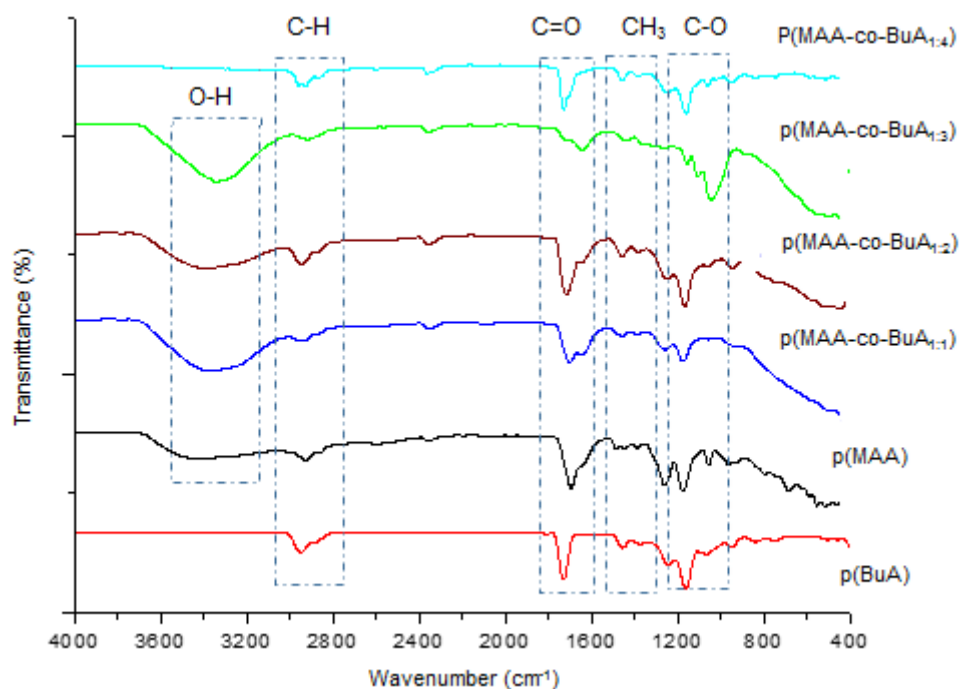
The emulsion polymerization of p(MAA-co-BuA) nanoparticles was conducted according to different molar ratios (Table 3). All the copolymer nanoparticles were obtained in white solid sticky form except for p(MAA-co-BuA<sub>1:4</sub>) which was obtained in

transparent plastic-like solid form (Figure 2(d)).

### Fourier Transform Infrared (FTIR) Spectroscopy

The formation of polymer nanoparticles was confirmed by Fourier transform infrared spectroscopy (FTIR). Figure 3 shows the comparison of IR spectra between p(MAA) and p(BuA) homopolymers with p(MAA-co-BuA) copolymers of different ratios of butyl acrylate (BuA) monomers.

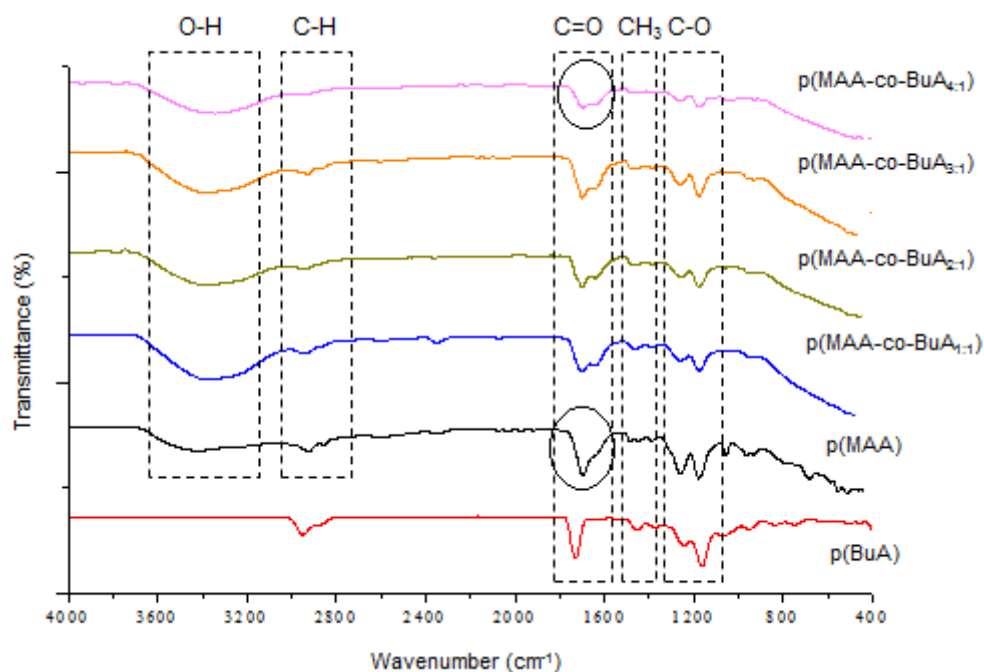
The presence of broad peaks which represents hydrogen bonded of hydroxyl group (OH) was observed in the range of 3000 – 3500 cm<sup>-1</sup> for all polymer nanoparticle samples except for p(BuA) nanoparticles, which do not comprise of OH group in the structure. The peaks obtained within the range of 2800 – 3000 cm<sup>-1</sup> belonged to the CH bond stretching, while the peaks in the range of 1450 – 1460 cm<sup>-1</sup> were due to the CH<sub>3</sub> stretching. The bands in the region of 1650 – 1750 cm<sup>-1</sup> belonged to the C=O bond functional group and the peaks obtained in the range of 1000 – 1300 cm<sup>-1</sup> were attributed to the C-O bond functional group.



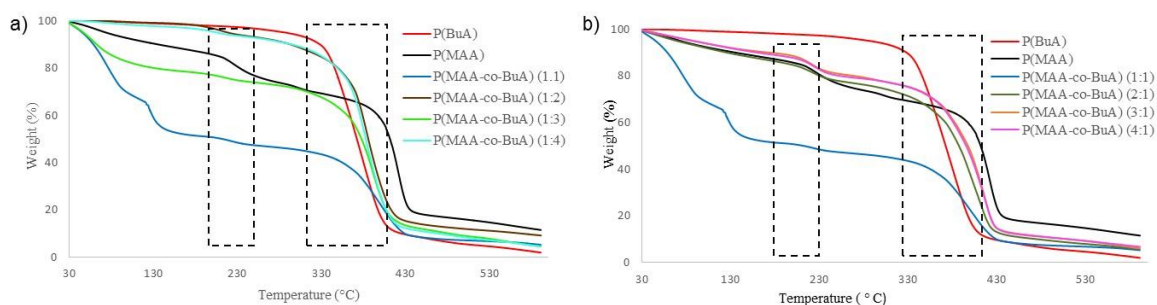
**Figure 3.** FTIR spectra of p(BuA), p(MAA), p(MAA-co-BuA<sub>1:1</sub>), p(MAA-co-BuA<sub>1:2</sub>), p(MAA-co-BuA<sub>1:3</sub>) and p(MAA-co-BuA<sub>1:4</sub>).

Figure 4 shows the comparison of both p(MAA) and p(BuA) homopolymers with the series of p(MAA-co-BuA) copolymers of different ratios of methacrylic acid (MAA) monomers. The IR band assignments for hydroxyl group (OH), CH and CH<sub>3</sub> stretching,

C=O and C-O groups were almost similar to p(MAA-co-BuA) copolymers of different ratios of butyl acrylate (BuA) monomers. As the ratio of MAA increased, the C=O became less intense, indicating the formation of p(MAA-co-BuA) nanoparticles.



**Figure 4.** FTIR spectra of p(BuA), p(MAA), p(MAA-co-BuA<sub>1:1</sub>), p(MAA-co-BuA<sub>2:1</sub>), p(MAA-co-BuA<sub>3:1</sub>) and p(MAA-co-BuA<sub>4:1</sub>).



**Figure 5.** Comparison of the TGA curves for p(BuA), p(MAA) and p(MAA-co-BuA)<sub>1:1</sub> with (a) p(MAA-co-BuA)<sub>1:2</sub>, p(MAA-co-BuA)<sub>1:3</sub> and p(MAA-co-BuA)<sub>1:4</sub>, and (b) p(MAA-co-BuA)<sub>2:1</sub>, p(MAA-co-BuA)<sub>3:1</sub> and p(MAA-co-BuA)<sub>4:1</sub>.

### Thermogravimetric Analyzer (TGA) Analysis

Thermogravimetric analyzer (TGA) analysis for the homopolymer and copolymer nanoparticles was performed to determine the weight loss of the polymer nanoparticles as a function of temperature. The thermal degradation behaviour of the polymer nanoparticles was studied in the range of 30 – 600°C. Poly(butyl acrylate) (p(BuA)) nanoparticles showed weight loss at approximately 332°C (Figure 5(a) and (b)). The major decomposition is assumed due to the degradation process of p(BuA) that occurs via the ester group scission. The thermal degradation of p(BuA) is expected to produce from small amount of monomers, while increasing the temperature will lead to the decomposition of the ester group, thus yielding products such as butane and anhydride [13].

The first weight loss of poly(methacrylic acid) (p(MAA)) nanoparticles was observed around 193°C and the second weight loss was at 401°C (Figure 5(a) and (b)). The major reason for the weight loss is assumed due to the production of water as a result of anhydride formation that dominates the minor decarboxylation reaction process [14].

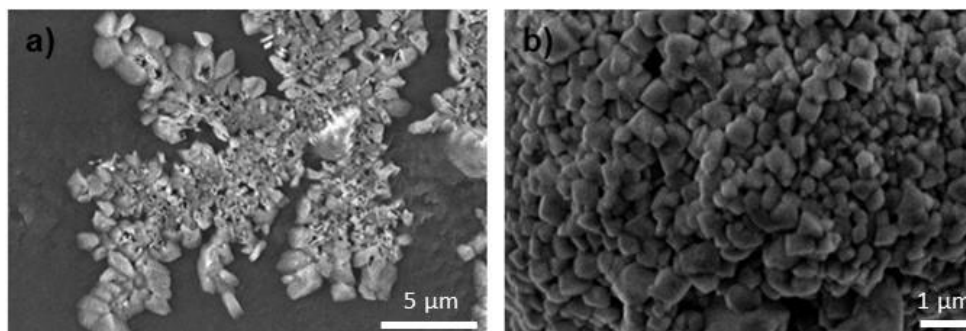
The multistage TGA curves which show the comparison between p(MAA-co-BuA) copolymer nanoparticles of different ratios of BuA monomers are depicted in Figure 5(a). The TGA curves for p(MAA-co-BuA) nanoparticles show there are two significant stages of degradation that occurred in the range of

180-350°C. Figure 5(b) shows the main stages of both homopolymers and p(MAA-co-BuA) nanoparticles degradation rates in different ratios of MAA monomers. The TGA curves for p(MAA-co-BuA) nanoparticles show there are two significant stages of degradation that occurred in the range of 180-380°C.

According to the study performed by Bajaj and his colleagues (1994) [15], the first step of weight loss of the copolymers nanoparticles in the range of 170 – 280°C may be due to the elimination of side products in the process of anhydride formation. The second weight loss observed in the range of 300 – 470°C may be attributed to the regions of major weight loss and caused by extensive degradation of the polymer backbone chain.

### Scanning Electron Microscopy (SEM)

The synthesized p(MAA), p(BuA) and p(MAA-co-BuA) nanoparticles were characterized by scanning electron microscopy (SEM) to determine their average particle size and surface morphology. The average particle size was obtained by randomly measuring the size of each particle obtained from SEM images (by referring to the scale bar), and calculating the average of all measured particles. The average particle size for p(MAA), p(BuA) and p(MAA-co-BuA) (Figure 6 and Figure 7) is in the range of 200 – 900 nm. Different shapes of particles such as spherical shape, rod-like shape and porous shape were observed.



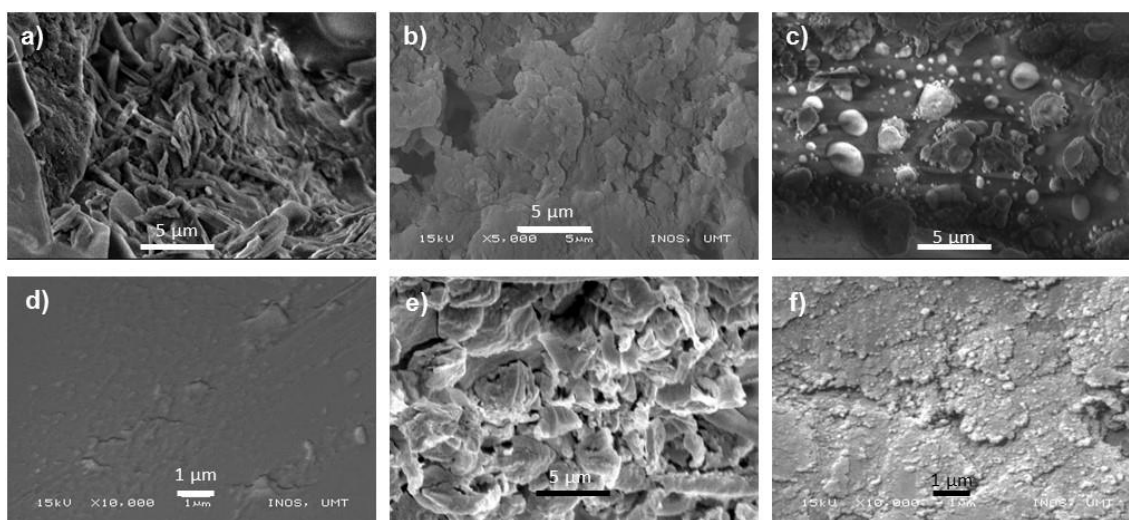
**Figure 6.** The morphology of (a) p(MAA) (5000x) and (b) p(BuA) (11000x) with scale bar 5 µm and 1 µm, respectively.

The morphology of p(MAA) and p(BuA) nanoparticles showed spherical-like shapes with average particle size of 880 nm and 260 nm, respectively (Figure 6(a) and (b)). The morphology of p(MAA-co-BuA<sub>1:1</sub>) (Figure 7(a)) showed rod-like shapes, while p(MAA-co-BuA<sub>1:2</sub>) (Figure 7(b)), p(MAA-co-BuA<sub>2:1</sub>) (Figure 7(d)) and p(MAA-co-BuA<sub>3:1</sub>) (Figure 7(e)) were in irregular shapes. p(MAA-co-BuA<sub>1:3</sub>) (Figure 7(c)) and p(MAA-co-BuA<sub>4:1</sub>) (Figure 7(f)) were in spherical-like shapes and their particle size were 690 nm and 360 nm, respectively. The degree of irregularity in particle shape of these copolymers may be caused by agglomeration

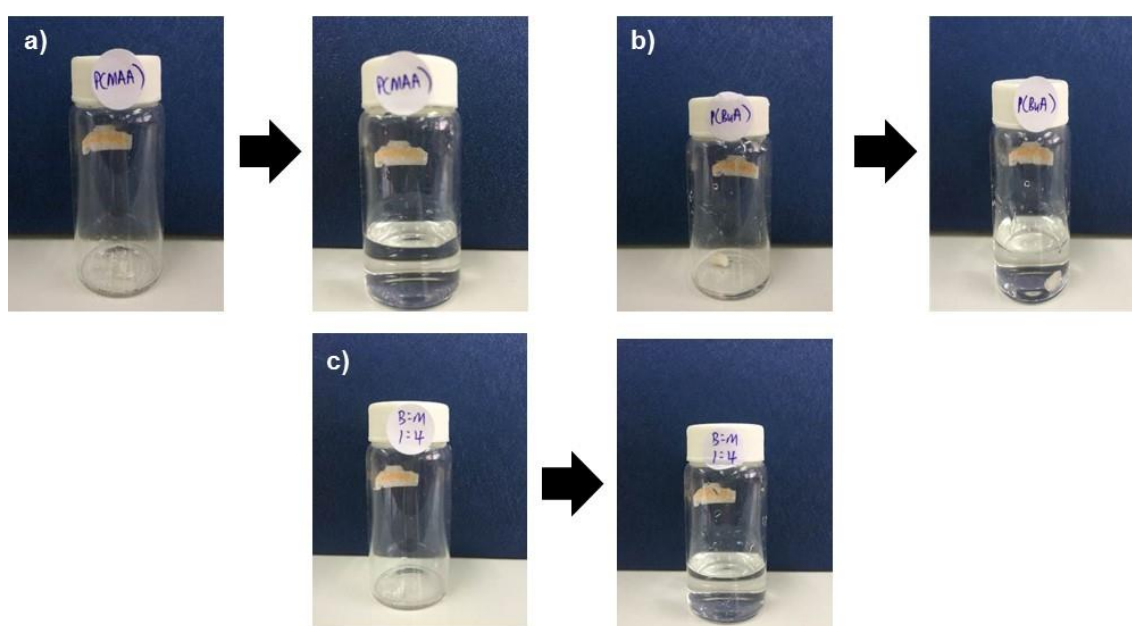
and fusion of particles upon coating and drying during the SEM sample preparation [16]. Therefore, it can be suggested that different molar ratios of methacrylic acid (MAA) and butyl acrylate (BuA) can affect the morphology of p(MAA-co-BuA) nanoparticles.

### Solubility test

Solubility test of p(MAA), p(BuA) and p(MAA-co-BuA) nanoparticles has been carried out to determine the degree of solubility of these nanoparticles. Figure 8 shows the result of the solubility test for each polymer nanoparticles.



**Figure 7.** The morphology of (a) p(MAA-co-BuA<sub>1:1</sub>) (5500x), (b) p(MAA-co-BuA<sub>1:2</sub>) (500x), (c) p(MAA-co-BuA<sub>1:3</sub>) (5000x), (d) p(MAA-co-BuA<sub>2:1</sub>) (10 000x), (e) p(MAA-co-BuA<sub>3:1</sub>) (5000x) and (f) p(MAA-co-BuA<sub>4:1</sub>) (10 000x) with scale bars 5 µm, 5 µm, 5 µm, 1 µm, 5 µm and 1 µm, respectively.



**Figure 8.** The solubility test of (a) p(MAA), (b) p(BuA) and (c) p(MAA-co-BuA<sub>4:1</sub>).

Based on the solubility test, p(BuA) nanoparticles (Figure 8(b)) were not soluble in distilled water due to its hydrophobic nature, while p(MAA) (Figure 8(a)) took approximately 4 minutes to dissolve in the aqueous medium. Increasing the molar ratio of butyl acrylate (BuA) or methacrylic acid (MAA) did not show any improvement in hydrophilicity of the polymer nanoparticles. However, p(MAA-co-BuA<sub>4:1</sub>) (Figure 8(c)) did dissolve in the aqueous medium but took approximately 6 min to completely dissolved in the distilled water. Hence, it can be suggested that p(MAA-co-BuA) nanoparticles of the ratio of 4:1 was obtained as the optimum ratio that has shown improved hydrophilicity by observing its solubility in aqueous solution.

### CONCLUSION

P(MAA), p(BuA) and a series of p(MAA-co-BuA) copolymer nanoparticles of different molar ratios of MAA:BuA (1:1, 1:2, 1:3 and 1:4) and BuA:MAA (1:1, 1:2, 1:3 and 1:4) were successfully synthesized by emulsion polymerization technique. The formation of the polymer nanoparticles was confirmed by FTIR where the absorption spectra showed similar peaks in all samples of the polymer nanoparticles. The TGA curves of p(BuA) nanoparticles showed significant weight loss at 332°C, while p(MAA) nanoparticles observed two significant weight loss at 193°C and 401°C. The multistage mass change for p(MAA-co-BuA) copolymer nanoparticles of different ratios of BuA and MAA monomers depicts two significant stages of degradation that occurred in the ranges of 180 – 350°C and 180 – 380°C. The SEM images presented the irregular morphologies of the polymer nanoparticles. The SEM images of p(MAA) and p(BuA) nanoparticles showed spherical-like shapes with average particle size of 880 nm and 260 nm, respectively. Due to the irregular structure and shape of the copolymer nanoparticles, only the particle size of p(MAA-co-BuA) nanoparticles of the ratios of 1:3 and 4:1, which showed spherical-like shape, could be calculated where their average particle size were 690 nm and 360 nm, respectively. For the solubility test, it was found that p(MAA-co-BuA) nanoparticles of the molar ratio of 4:1 was obtained as the optimum ratio that showed improved hydrophilicity of butyl acrylate. Therefore, it can be concluded that the copolymerization between MAA and BuA could produce improved hydrophilicity of p(MAA-co-BuA) nanoparticles.

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