

Using Microwave as a Powerful Tool in Polymer Science

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Abstract: Microwave is a powerful heating source that is using a unique principle for heating. The heat is generated internally which means all the polymer parts will get same amount of heating at almost the same time. This heating technique is very useful in polymer synthesis and polymer waste treatment. In polymer synthesis, equal heating will unify the degree of polymerization resulting much less degraded polymer parts and unpolymerized monomers. In polymer waste treatment, unified heating is preferred in pyrolysis process. This review is discussing the impact of microwave on the polymer science.

Keywords: Microwave, Polymer, waste treatment, polymer synthesis, and Graft polymerization.

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I. Introduction

Polymers are one of the greatest inventions in the 20th century[1]. They can be found almost everywhere in daily life such as: plastics, packaging, insulating, painting, and textile fibers[2, 3]. Global markets have an increased demand on polymers with growing attention to save the environment[4]. Saving the environment can be done through more efficient polymerization methods and polymer waste treatment[5]. Effective polymerization methods will reduce polymer waste during polymerization and the polymer cost. The annual global plastic production is about 360 million tons[6]. The major part of these plastics is not biodegradable which means after it used, these plastics will accumulate as polymer waste in land and sea[7]. Polymer waste is a huge problem that threatens the eco system. It estimated that the amount of plastic waste about 7 billion tons[8]. Polymer waste treatment is a powerful way to reduce pollution and save the environment. There are many polymerization and waste treatment methods, but heating is one of the most common methods. Equal heat distribution can lead to proper polymerization and eventually less waste. In addition, the equal heat distribution is very useful to degrade polymers back to monomers which is essential to recycling process.

Heating is one of the most used methods for polymer synthesis as well as curing and post curing. Heating is also important to plastic recycling process. Conventional heating process is based on a high temperature heating source in direct contact with the polymer surface via conduction.[9] Many polymers have low heat conductivity which will affect heat distribution from the polymer surface to the other parts of the polymer[10]. Unequal heat distribution means unequal polymerization degree, curing, nor post curing. The polymer surface will have remarkably elevated temperature which might lead to degradation or other unwanted side reactions[11]. At the same time, the temperature is decreasing from the surface toward the polymer core which might led to less degree of polymerization or no polymerization.

On the other hand, microwave is a powerful internal heating method. It uses radio frequency to generate heat from the non-conductive polymer itself[12]. Microwave generates heat internally from the polymer itself through dielectric relaxation. Internal heating has even heat distribution through entire polymer. The temperature can be controlled by controlling irradiation time and power[13]. These polymers dissipate radio waves as heat form by dielectric relaxation and reversing the polarization continuously. Microwave assisted heating has many advantages over conventional heating[14, 15]. Microwave has equal heat distribution, faster heating, less energy, in most cases it does not need solvents, less waste, more economic, and it is ecofriendly[16-21].

Microwave reactors have been used before 1990[22]. A breakthrough occurred in microwave reactors technology after 1990 in terms of using closed pressure reactors as well as temperature and pressure controlled

equipment[23]. The temperature can be controlled using IR controlled pyrometer or fiber optic temperature sensor. The microwave became more trusted technology due to high reaction replication. Microwave reactors can be classified into two main classes: monomode and multimode reactors[24]. The monomode microwave irradiate only one vessel while the multimode irradiate more than one vessel at the same time.

This review focuses on using microwave in polymer synthesis, grafting of polymers, and polymer waste treatment.

II. Polymer synthesis

2.1 Addition polymerization

Addition polymerization is one of the most common polymerization methods. It is used for unsaturated monomers. This type of polymerization is initiated by reactive species such as free radicals, anions, and cations to open the π bond which will turn that monomer to a radical, anion, or cation respectively[25]. Then another monomer will be added to first monomer and the chain will start to grow till it stopped by adding a material that destruct the reactive center. Polypropylene, polyvinyl chloride (PVC), polyethylene, and polystyrene are the most common addition polymers.

Microwave have been used in ring opening polymerization, step-growth polymerization, and addition polymerization[26-29]. An exponential interest of using microwave in addition polymerization for both of the scientific and industry purposes. The advantages of using this type of heating over the conventional heating is faster polymerization, molecular weight consistency, reproductivity, and less polymer side products.

A domestic microwave oven was used efficiency for bulk-polyaddition of styrene, acrylonitrile, vinyl acetate, and methyl methacrylate monomers[30]. In addition to catalytic amounts from radical initiators Azobisisobutyronitrile (AIBN) or benzoyl peroxide (BP). Conversion percentage was ranged from 65-99 % in shorter time. Furthermore, lowering the power with shorter time gives higher molecular weight and homogeneous polymers. In another study, microwave irradiation was used for methyl methacrylate (MMA) polymerization and radical initiators [31]. In addition to two radical initiator systems, AIBN/CuBr₂/Bipyridine (bpy) and AIBN/CuCl/bpy, were applied. The results showed that the microwave improved polymerization rate, narrowed the polydispersity index, and decrease the amount initiator dramatically when it is compared with conventional heating.

Fumaric esters are one of addition monomers (figure 1). Polymerization of these monomers in conventional heating methods is very slow because of the steric hinderance of the alkyl groups. It is found that applying microwave irradiation for polymerization of diisopropylfumarate (DiPF) enhance the polymerization rate dramatically[32]. Three different irradiation powers were used: 140, 210, and 280 W with two radical initiators: AIBN and BP. As the irradiation power increases, the monomer conversion increases drastically up to 10 minutes and BP has higher conversion rate than AIBN.

Polyacrylamide (PAM) is a water soluble polymer. It is used in wide range of applications such as gel electrophoresis, paper industry, and treatment of sludge[27]. Some of the PAM applications require specific molecular weight and types of the functional group on sidechains. To achieve this goal, some methods use inert atmosphere and specific catalysts. However, yield still low ~ 50% and some of the products are colored. When the microwave is used to prepare the PAM, yield elevated to 98.5% and the product molecular weight was between 4×10^4 to 1.3×10^5 . The molecular weight was highly controlled by the microwave power.

2.2 C-C coupling polymerization

Forming carbon-carbon bond is very important for carbon building blocks[33]. It is used for building aliphatic and aromatic molecules as well as polymers. Aryl-aryl bond can be formed through many reactions by using aryl halide and a metal or other methods[34]. Ullmann coupling is one of well-known reactions to form carbon-carbon using aryl halides and copper, Nickel, and Palladium[35].

Poly(p-phenylene) is used in many applications such as: solid-state laser materials, two photon devices, and light-emitting diodes[36, 37]. These polymers often called ladder polymers. Polymerization of phenylenes require a polyketone precursor that is subjected to two steps of polymer analogue. That is followed by reaction with methyl lithium to form a tertiary alcohol and the tertiary alcohol is cyclized in presence of Lewis acid. When conventional heating is used, it is hard to optimize the experimental conditions to produce conjugated polymers with high molecular weight. In addition, the polymerization time will be long. Nehls et al. reported synthesis precursor of 2,6-naphthylene polyketone (**2,6-NPK**) (figure 2-A), 1,5-naphthylene polyketone (**1,5-NLP1**) (figure 2-B), and (**1,5-NLP2**) (figure 2-C) using microwave reactors. **2,6-NPK** can be polymerized

Figure

(1): diisopropylfumarate

(2): BP

through conventional and microwave while ,5-NLP1 and ,5-NLP2 can be polymerized only by microwave. In another study, Poly(9,9-dihexylfluorene)s (PDHFs)

A B C

Figure (2): A:2,6-naphthylene polyketone (2,6-NPK) structure, B:1,5-naphthylene polyketone (1,5-NLP1) structure, and C:1,5-naphthylene polyketone (1,5-NLP2) structure

were prepared using microwave[38]. It took 14 minutes to get 40,000 molecular weight in comparison with 48 hour to get 20,000 in conventional heating. The study showed that longer microwave irradiation times produces unwanted crosslinked polymers. Li and coworkers study 3 homopolymers PPDI(H), PPDI(OD), and PNDI(OD) (figures 2-A, 2-B, and 2-C) respectively in both of conventional and microwave heating[39]. When the oil bath is used, no product was

A B C

Figure (3): A: PPDI(OD) structure, B: PPDI(H) structure, and C: PNDI(OD) structure

obtained for 80 C and 12 hour reaction time. When the temperature and time was raised to 150 C and 24 hours, only 6 repeating unites were produced. While the microwave irradiation produce 11 repeating unites in just 5 minutes.

III. Graft polymerization

Incorporating monomers into a polymer backbone and polymerization of these monomers is called grafting polymerization[40]. It is one of the methods to blend two different polymers with wide range of functional groups. Grafting process is initiated by ionizing radiation, gamma radiation, electron beam, and microwave irradiation. These polymers are used in textiles, separation, purification, coatings, laminates, adhesives, and wastewater treatment.

Wastewater treatment is very important to reduce pollution and reuse the water. As the global population increases, the urban, agricultural, and industry wastewater increase dramatically. There are many wastewater treatment methods such as: extraction, adsorption, coagulation, activated carbon, precipitation, evaporation, oxidation, membrane filtration, ion-exchange, and biodegradation[41]. Usually, more than one method is used to better remove the contaminations. The coagulation-flocculation method is one of the most commonly used[42]. The waste water treatment process has two main stages: first, rapidly mixing the coagulants, and second, using gentle agitation to flocculate the agglomerated small particles. Then the flocs are removed after it settle down as sludge.

Natural and synthetic polymer flocculants are well known due to their biodegradability, low-cost, non-toxicity, and shear resistance. Pal et al report synthesis of polyacrylamide (PAM) grafted dextrin (Dxt-g-PAM) assisted by microwave irradiation[43]. They found that higher and bigger gyrate radius has the best performance. Even the synthesized grafted polymer was better than the commercial flocculant 226 LV. In another study, Mishra et al test PAM grafted starch (St-g-PAM) using conventional chemical and microwave assisted grafting polymerization[44]. The grafting polymer produced with microwave assistance was better conventional chemical method in terms of synthesis time, reproductive, more reliable, and higher quality.

Microwave initiated grafting polymerization of Psyllium husk and PAM (Psy-g-PAM) without any radical initiator was reported by Sen and his coworkers[45]. Variable irradiation times and acrylamide monomer concentrations resulted variable grades of Psy-g-PAM. The optimal conditions that produced higher grafting and best flocculation were 1 minute irradiation and 0.2 g/mL monomer concentration. The microwave assisted grafted polymerization of gum gatti PAM (GGI-g-PAM) using ceric ammonium nitrate (CAN) as radical initiator was reported by Rani and coworkers[46]. A few grades of grafted GGI-g-PAM were prepared. Half of the grades have different CAN and the other half have the acrylamide different concentration. Keeping

the microwave irradiation time at 120 seconds mainly and 1 g of gum gatti. The best grafting was obtained with 0.4g of CAN and 5g of acrylamide.

Grafted PAM on β -cyclodextrin was synthesized in five different grades by using microwave assisted (β -CD-g-PAM-MA) and solution (β -CD-g-PAM-S)[47]. These five grades were evaluated as flocculants by two methods. The β -CD-g-PAM-S3 and β -CD-g-PAM-MA4 showed the best flocculation performance. Fenugreek gum grafted PAM (FG-g-PAM) was reported by Mishra and his coworker[48]. The grafted polymer was prepared using thermal and microwave assisted methods. The flocculation, deflocculation, and reflocculation of these polymers were examined. The best flocculation efficiency was ranged from 64.94% to 95.98%. In addition, they found that the flocculation of the grafted polymers increases after reflocculation process up to 95.98%.

IV. Polymer waste treatment

Polymer production has increased dramatically over the years which led to increasing of the polymer waste. A large part of this waste is accumulated in the land and sea causing many hazards to the environment[49]. There are many polymer waste treatment methods (e.g. incineration, energy recovering systems, recycling, and pyrolysis). Incineration is not desirable because it produces many harmful gases. Recycling also has some drawbacks like high cost and water contamination. Energy recovery is a better option in terms of environmental and energy supply. Pyrolysis uses heat up to 500 C without oxygen to degrade the polymer. In pyrolysis heat is transferred from the polymer surface to the core via thermal conductivity. The heat distribution will be different from layer to layer inside the polymer which will produce a wide range of degradation products. This will limit the usage of the degradation products. Microwave assisted pyrolysis is another approach to incorporate the microwave to pyrolysis process[7]. Microwave heating is much more evenly distributed which will lead to much narrow pyrolysis products. The products type can be controlled via microwave power and time[50]. High power will favor gas production as the power drops down as the product will change to liquid then to solid.

Aishwarya et al. reported microwave assisted pyrolysis of various plastic types. They use 2.45 MHz microwave reactor with up to 5 kW power. In addition to quartz reactor, cold trap, and condensers. The products of the microwave assisted pyrolysis (MAP) were analyzed using FTIR, TGA, and GCMS techniques. The GCMS showed various acids such as decadionic, octadecanoic, and hexadecenoic acids. Undri and coworkers studied the MAP reverse polystyrene polymerization back to styrene[51]. The microwave power was 3 kW and the amount of polystyrene used was 100g. The pyrolysis gave low viscosity clear liquid with overall yield was 87%. The GCMS show the products were: aromatic compounds (C6-C10) 94%, styrene 66%, char 10%, and gas 4%. In similar study, Bartoli et al. studied the depolymerization of polystyrene under reduced pressure in presence or absence on nitrogen gas[52]. The effect of heating and pressure rates on the amount of the produced liquid were examined. Different heating and pressure rates were applied. The highest amount of liquid (94 wt%) was obtained in presence of nitrogen gas with 28 minute reaction time. Rosi et al. studied the MAP of halogenated plastics from end-life computers[53]. They amount of liquid formed from the pyrolysis was up to 77%. The liquid was characterized by FTIR, NMR, and GCMS. It has some useful materials such as styrene and xylene. In addition, it has low viscosity and density

V. Conclusion

Microwave is very useful internal heating tool. Heat distribution in microwave is better than any other heating method. The yield is mostly higher, and time is much shorter. Microwave have been efficiency used in polymer synthesis and polymer waste treatment. In some cases, polymer synthesis or polymer pyrolysis was only done through microwave irradiationit.

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