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Batch & Semi-Continuous Process Effect on Conversion of Methyl Methacrylate and Butyl Acrylate Copolymer By Suspension Copolymerization

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Abstract: ***Batch and Semi-continuous Suspension Copolymerization of Methyl Methacylate (MMA) with Butyl Acrylate (BA) was carried out in four neck three liter round bottom flat flanged glass reactor, in presence of Azobisisobutyronitrile as initiator, Potassium salt of MMA & Polyvinyl alcohol as stabilizer and Toluene as solvent. Experiments were carried out to study the effect of batch and semi-continuous process on conversion of copolymer at different reaction temperature. The value obtained of conversion was higher in semi-continuous process than batch process.***

Keywords: Suspension, Copolymer, Methyl methacrylate, Butyl acrylate, Conversion, Batch, Semi-continuous

1. Introduction

Suspension polymerization method is extensively used today for polymer to obtain polymer of choice in terms of molecular weight, structure, and crystallinity. Suspension polymerization is offering a good dissipation and control on the heat of polymerization by the medium of suspension water and at the same time completing the polymerization because of the small sphere reactor which is within the small bead. [1]

The reaction is generally ended with the small beads which are easily dried to obtain an excellent product. Suspension polymerization refers to polymerization in an aqueous system with monomer as dispersed phase, resulting in polymer as a dispersed solid phase. Only water insoluble monomer can be polymerized by this technique. [2]

Suspension polymerization is used since it offers better temperature control, little or no gel effect and therefore better molecular weight control with 95 to 100 % conversion easily. Suspension polymerization is also useful than other polymerization techniques in case of optical plastics such as PMMA, PS, PSMMA, of course P(MMA-BA). [3]

In spite of a large literature, many parameters are insufficiently, disclosed and much additional research could be done. In general, no data are available directly on copolymerization of Methyl methacrylate and Butyl acrylate. Suspension polymerization of Methyl methacrylate-Butyl acrylate copolymer was investigated to find out effect of batch and semi-continuous process on conversion at different reaction temperature.

2. Experimental

A. Materials and Chemicals:

Monomer:

1) Methyl Methacrylate (MMA) contained less than 100 ppm hydroquinone as inhibitor was removed by extraction with 0.1 N NaOH followed by water wash.
2) Butyl Acrylate (BA) of 99% purity as specified by manufacturer was used as received.

Initiator: Azobisisobutyronitrile
Stabilizers: Potassium salt of MMA and Polyvinyl Alcohol
Continuous Phase: D.M. Water
Solvent: Toluene
Methanol was used for removal of unreacted monomer.
Nitrogen: Nitrogen was purged to remove oxygen from reactor which inhibits the polymerization.

B. Experimental Procedure

Batch and Semi-continuous suspension copolymerization was carried out in four neck three liter round bottom flat flanged glass. Through center neck an agitator was fitted in to the reactor. The other necks were used to fit the thermometer pocket, sampling tube and condenser. The stirrer was drives by ½ HP motor. [4]

First, the required amount of double distilled water and PVA and Potassium salt of MMA charged to the reactor. The reactor was then heated to the required temperature 70 ^{o}C to 80 ^{o}C at constant temperature bath with stirring facility to keep the uniform temperature in the bath. The agitator was also started. Sufficient time was allowed (Approx. 45 min.) for temperature to reach the required temperature and to ensure the complete dissolving of PVA in the distilled water.

The monomers used contain inhibitor which was washed with 0.1 N NaOH solutions then it was washed with water for five to six times for removal of NaOH. In a separate container required amount of inhibitor free Methyl

Methacrylate (MMA) and Butyl Acrylate (BA) were taken and required amount of initiator Azobisisobutyronitrile (AIBN) was added to the mixture of two monomer. After one hour (when temperature was reached to desired value) the mixture of monomers were added to the reactor through a nozzle by dropping funnel for batch process and for the semi-continuous process the mixture of monomers and initiator were added to the reactor though a nozzle for throughout the reaction time semi-continuously.

It was essential that the stirrer should not be stopped from the time the BA and MMA has been charged until the end of polymerization run, otherwise the reaction mixture gels. After 6-7 hrs (total reaction time) heating was stopped and cooling water was charged from the top of water bath and hot water removed from the bottom of water bath. After that stirrer was stopped, whole mass was taken out from the reactor to one lit beaker. Water was filtered off to isolate polymer beads. These beads were washed repeatedly with warm water for six times to remove adsorbed PVA from beads. The washed beads were finally immersed in 200 ml of methanol and stirred for 30 min. to remove unreacted monomer. The samples were dried to constant weight in a vacuum oven at 60 ^{o}C.

Experiments were performed for three different temperatures (70 ^{o}C, 74 ^{o}C, 80 ^{o}C) and constant initiator concentration (0.6%) for batch and semi-continuous manner. Other parameters were remains constant.
Standard recipes for above experiments were followed:
Monomer to water ratio: 1:3
MMA to BA ratio: 3:1
Suspension stabilizers concentration: 0.4% of monomer weight
Initiator concentration: 0.6% of monomer weight
Reaction temperature: 70 ^{o}C, 74 ^{o}C and 80 ^{o}C
Process: Batch and Semi-continuous

3. Analysis and Testing

Conversion:
Samples at fix time interval were taken out from reactor for determination of intermediate fractional conversion. Samples from the reactor were taken out with the help of widely open mouth pipette with rubber ball on the top. The pipette was cleaned after each sample withdrawn in order to remove the sticky mas inside the pipette which might get chocked and sampling would have been difficult for next interval. The sample takes out form the reactor was collected in a cylinder that contained 10 ml toluene.

The sample taken was analyzed till the next sample withdrawn. In order to determine the progress of polymerization reaction sample were taken out at every fix time interval from the start of polymerization reaction.

4. Results and Discussion

Effect of batch and semi-continuous process on conversion:

Table 1: Experimental Runs

Experiment No.	Reaction Temperature (^{o}C)	Types of Process	Conversion (%)
1	70	Batch	87
2	74	Batch	89
3	80	Batch	91
4	70	Semi-Continuous	88
5	74	Semi-Continuous	90
6	80	Semi-Continuous	94

Fig. 1 shows for 0.6% concentration and different temperature the effect of batch process on conversion. It shows that if temperature increases at same initiator concentration the conversion were achieved higher values in batch process

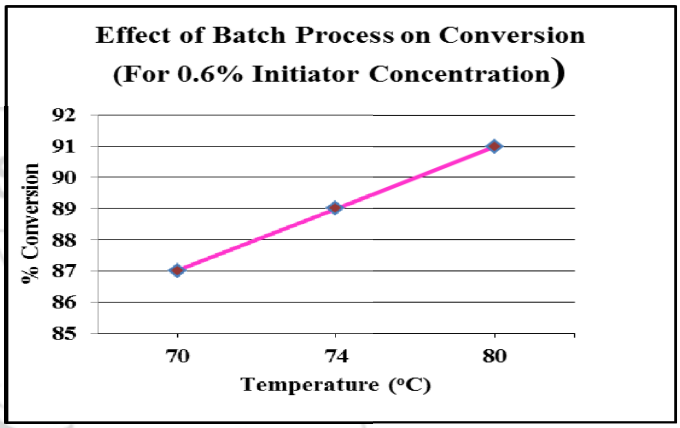

Figure 1: Effect of batch processs on conversion for different temperature (For 0.6% Initiator Conc.)

Fig. 2 shows 0.6% concentration and different temperature the effect of semi-continuous process on conversion. It shows that at same initiator concentration the conversion were achieved higher values in batch process

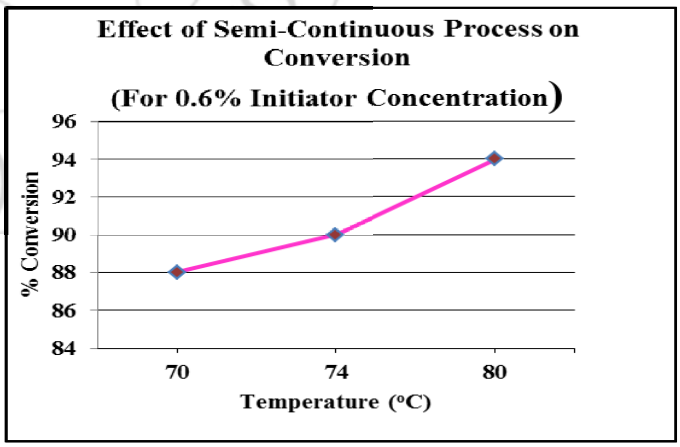

Figure 2: Effect of semi-continuous processs on conversion for different temperature (For 0.6% Initiator Conc.)

Fig. 3 shows (for 0.6% concentration) and three different temperatures the effect of batch and semi-continuous process on conversion. It shows that the conversion increase in semi-continuous process with increase in temperature with same initiator concentration.

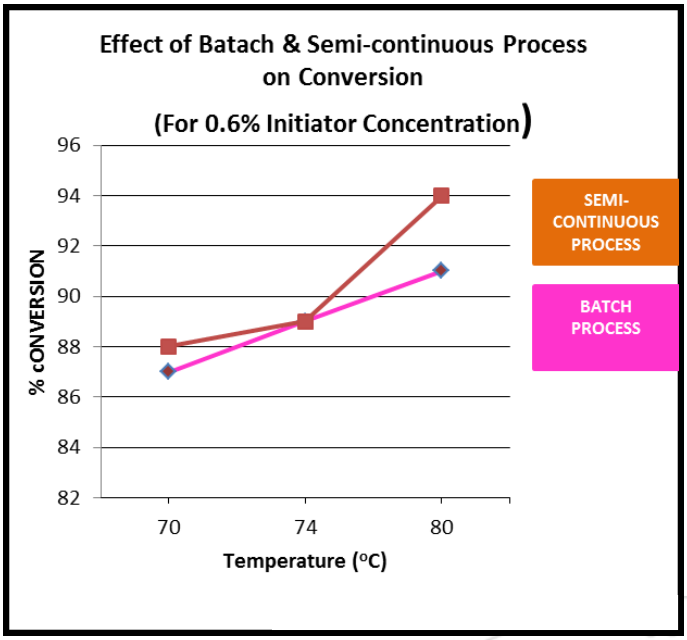

Figure 3: Effect of batch processs and semi-continuous process on conversion for different temperature (For 0.6% Initiator (Conc.)

5. Conclusion

From figures and laboratory synthesis of Methyl methacrylate and Butyl acrylate suspension copolymerization, with different temperature and same initiator concentration, it was observed that, higher conversions were obtained at higher temperature for semi-continuous process. At 80 °C temperature and 0.6% initiator concentration 94% conversion was obtained.

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