

Particle Size and Particle Size Distribution of Suspension Polymerization of Methyl Methacrylate in Baffled Tank Reactor

R.M. Mohsen and Y.M. Abu-Ayana

Polymers & Pigments Department, National Research Centre, Cairo, Egypt.

SUSPENSION polymerization is commonly used to produce micron-sized (10-100 μm) polymer beads, in which the final particle size distribution is an important end-use property. A laboratory reactor was designed and constructed in the author's previous article to study the effect of both speed of agitation and concentration of suspension stabilizer on particle size and particle size distribution during the suspension polymerization of methyl methacrylate. In this article, scaling up of the laboratory reactor was done in order to find out the effect of change of the baffled tank reactor dimensions on the relation between particle size and both speed of agitation and stabilizer concentration. It was found that there were contradictions of the effect of speed of agitation on the particle size from the previous results. New empirical equations correlating the average particle size and particle size distribution (PSD) were derived from this study.

Keywords: Suspension polymerization, Poly methyl methacrylate (PMMA), Polystyrene (PS), Polyvinyl chloride (PVC), Particle size distribution (PSD).

Suspension polymerization processes ⁽¹⁾ are commercially important for the production of polymer beads having wide applications such as chromatographic separations and ion-exchange resins. A wide spread fragments applicability of microparticles in different fields ⁽²⁾ including medical, textile, cosmetics, pesticide, printing industry and pharmaceutical. Novel molecularly fragments imprinted polymers –membranes, microspheres, and photoswitchable particles ⁽³⁾. Microporous polymer beads for chemical decontamination ⁽⁴⁾. The particle size distribution (PSD) may appreciably influence the performance of the final product; therefore, the evaluation of (PSD) is a major concern in the design of a suspension polymerization process. The most important factors in the practical operation of suspension polymerization ⁽⁵⁾ is the control of the final particle size and particle size distribution. Many research studies have indicated that the particle size distribution is a result of the dynamic equilibrium between breakage and coalescence of the monomer droplets. An important limitation in the development of the microparticles is the difficulty to control particle size. Variation of droplet size during the whole polymerization process under the unsteady stirring condition, compared with that under a steady stirring condition, was found to be small ⁽⁶⁾. Some of the variables affecting the particle size (PS) and the particle size distribution (PSD) in suspension polymerisation ^(7,8) are :

1. Type, molecular weight, and concentration of suspension stabilizer.
2. Agitation rate and agitator design, reactor design, and position
3. Water-monomer
4. Reaction temperature
5. Reaction time, type, and concentration of the radical initiator
6. Type and concentration of monomer
7. Effect of additional surfactant, electrolyte, and/or viscosity improvement
8. Ratio of initial monomer viscosity to aqueous phase viscosity
9. The Reynolds and Webber Number.

Some of the technologies used in evaluating particle size are sieve analysis^(9,10), coulter counter, and microscopy. Polacco⁽¹¹⁾ studied the measurements of particle size distribution during suspension polymerization. A scanning electron microscope was employed to show the spherical nature of particles. Near infrared reflectance spectroscopy (NIRS) may be used efficiently for in-line evaluation of average particle size in suspension polymerization⁽¹²⁾. Measurement of particle distribution in suspension polymerization was done using in situ laser backscattering⁽¹³⁾.

A suspension polymerization process that generates narrow-size distribution and spherical particles in the range of 3–10 μ was described by Kamiyama⁽¹⁴⁾. Santos *et al.*⁽⁵⁾ also studied, the effect of agitation speed and stabilizer concentration on particle size distribution in laboratory scale styrene suspension polymerization reactors. Preparation of magnetic polystyrene with a narrow (PSD) was carried out⁽¹⁵⁾.

Parameters affecting the final particle size and the particle size distribution under unsteady stirring conditions, such as the agitation speed and the periodic interval also have been studied in details⁽¹⁶⁾ and the results were compared with those under a steady stirring condition. Suspension polymerization used for the production of polymer particles with diameters in the range of 50–750 μ m was studied⁽¹⁷⁾.

The particle size distribution of the particles produced in batch suspension polymerization was calculated using a population balance model^(18,19). Studies were carried out to develop a mathematical model based on experimental results to correlate agitation and other polymerization parameters with particle size of polystyrene^(6,15,20), polyvinyl chloride (PVC)⁽²¹⁾ and polymethyl methacrylate⁽¹¹⁾. Effect of mixing process on the (PSD) and the mean diameter on thiol-triacrylate microcapsules was studied⁽²⁾.

A baffled laboratory scale reactor (1/4 liter capacity) with a flat paddle agitator⁽²¹⁾ had been previously designed (by the present authors) to investigate the effect of the impeller speed and the suspension stabilizer concentration on the average PS and PSD of PMMA particles).

In this article, scaling up of the laboratory reactor to two liter capacity was done in order to find out the effect of the baffled tank size on the relation between speed of agitation and stabilizer concentration on PS and PSD.

Experimental

The baffled tank reactor of the same authors was enlarged to 2 liter with nearly the same geometrical dimensions to find out the effect of the speed of agitation and stabilizer concentration on PS and PSD.

Apparatus

A laboratory scale reactor (Fig. 1) was designed and constructed from stainless steel. It consisted of two parts, a cylindrical kettle of two liters capacity and a cover with a flat paddle agitator. The cover has four bolted upwards and downwards deflecting baffles, and two holes, one in which a glass condenser was fitted to condense any escaping monomer and the other in which a thermometer was fitted. The following geometric dimensions are fixed throughout the whole batches:

Hight of the tank H_t = diameter of tank D_t = 130 mm

Hight of tank H_t Hight of liquid H_L = 1.4

Diameter of impeller D_i = 90 mm

Blade W_i = 10 mm

Elevation of impeller above tank bottom Z = 31 mm

Width of baffles W_b = 10 mm

Number of baffles = 4

H_L / D_i = 1.4 D_t / D_i = 1.4

Z / D_i = 0.34 W_i / D_i = 0.11

W_b / W_i = 0.11

It must be realized that any change in the geometrical arrangement affects considerably the particle size.

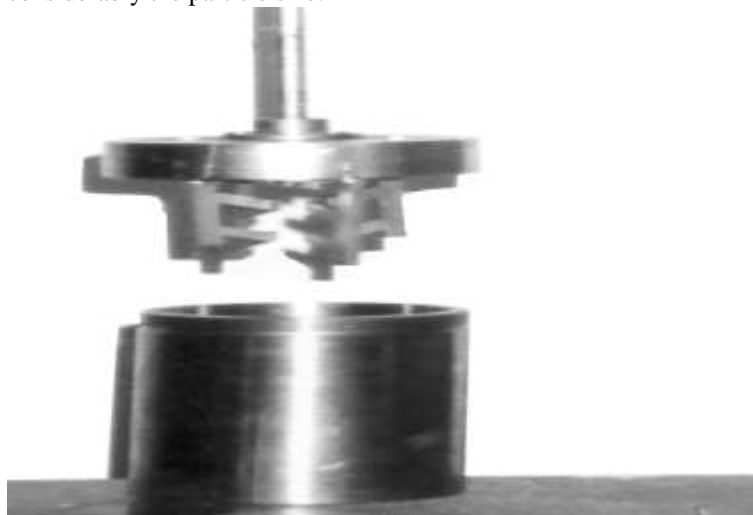


Fig. 1. Laboratory scale baffled reacto.

Technique

Distilled water and soluble starch stabilizer were charged in the kettle; which was put in a thermostatically controlled water bath at 80°C and the impeller was worked at the intended speed, N, which was measured periodically throughout the time of reaction by a speedometer. When the agitation became regular, methyl methacrylate monomer containing benzoyl peroxide was added. The reactant amounts are given in Table 1.

The time/temperature and speed of agitation were recorded periodically throughout the whole reaction. The gel stage was clearly observed by the rise of $\approx 5-8^\circ\text{C}$ above the constant reaction temperature. Much care should be taken at this stage, since the increase in viscosity may slow down the agitator causing the polymer particles to coagulate. After the gel stage has been safely accomplished, the temperature went down again to normal value such that no danger of coagulation since the polymer beads with their smooth surfaces had already been formed. After one hour, the reaction was nearly completed, heating and agitation were stopped, and the product was left to settle down followed by decantation of liquid, washing, and drying. A sieve analysis was performed to classify the product into grades of narrow PSD. The average PS of each experiment was determined from the sieve analysis according to method 1⁽⁹⁾.

Materials

The materials used, classified under water phase and monomer phase, are given in Table 1.

TABLE 1. Reactants amounts .

Water phase	Monomer phase
1106 gm water 15.25 gm Na ₂ HPO ₄ 0.406 gm NaH ₂ PO ₄ S gm soluble starch	578 gm MMA 2.899 gm BZ ₂ O ₂

S= variable amount of soluble starch.

Method 1⁽⁹⁾

Cumulative weight percent retained on is plotted against equivalent diameter of each two successive sieves on a log-log paper. The average particle size $D_{50\%}$ was determined; also $D_{16\%}$ and $D_{84\%}$ cumulative weight retained on were determined. The dispersion and skew were calculated according to the following equations:

$$\text{Dispersion} = (D_{84\%} - D_{16\%}) / 2$$

$$\text{Skew} = [(D_{84\%} - D_{50\%}) - (D_{50\%} - D_{16\%})] / (D_{84\%} - D_{16\%})$$

The following notations will be found:

X = weight fraction retained on each sieve.

% cumulative weight retained on sieves = X/ total weight.

D_{av} = equivalent diameter of each two successive sieves.

D_m = average particle size in accordance to measurements of the particle size by method1

$D_{16\%}$ = particle size at 16% cumulative weight retained on.

$D_{50\%}$ = average particle size at 50% cumulative weight retained on.

$D_{84\%}$ = particle size at 84% cumulative weight retained on.

PSD = particle size distribution.

N = speed of agitation in rpm

C = concentration of soluble starch

Results and Discussion

Factors affecting the gel stage

Though the suspension polymerization seems to be simple, the practical control of the reaction conditions is quite difficult, since tendency to coagulation of the globules is a considerable factor, which leads to the deterioration of the batch. The gel stage which can be considered the bottle neck in suspension polymerization reaction needs special care since the excessive heat of polymerization evolved at this stage produces a temperature rise of 5– 8°C above the normal reaction temperature. Agitation will help distributing this heat and would prevent local overheating.

Two sets of experiments were performed in order to study the effects of agitation rate and starch concentration on the average particle size and PSD of the product.

TABLE 2. Relation between the concentration of starch and gel formation time at 480 rpm .

% Concentration of starch	Time (min.)
0.75	75
1.00	75
1.25	80
1.50	75
1.70	75
1.756	75

TABLE 3. Relation between the concentration of starch and gel formation time at 580 rpm.

% Concentration of starch	Time of gel formation,(min.)
0.795	125
1.00	115
1.24	110
1.50	110

TABLE 4. Relation between the speed of agitation and gel formation time at 1% concentration of starch.

Speed of agitation (rpm)	Time,(min)
460	75
500	80
560	105
660	155

TABLE 5. Relation between the speed of agitation and gel formation time at 1.5% concentration of starch.

Speed of agitation (rpm)	Time, (min)
480	75
580	110
680	155
780	195

Tables 2 & 3, show that the gel stage formation time is independent of the concentration of starch, while Tables 4 & 5 represents the dependence of the time of attaining the gel stage on the speed of agitation at constant concentrations of starch. It is proportionally increase with speed of agitation.

There are relations between speed of agitation N and stabilizer concentration C on the particle size and particle size distribution.

The speed of agitation N (400-900 rpm) at constant concentration of starch (1-1.5% C) has an effect on PS and PSD.

Figures 2 & 3 show the cumulative plots retained on sieves against average diameter at 1 and 1.5 % starch concentration, at variable agitation rate.

From Fig. 2 and 3, Tables 6 and 7 were obtained which summarize the variation of $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, dispersion and skew with the speed of agitation N at 1 and 1.5% C , respectively.

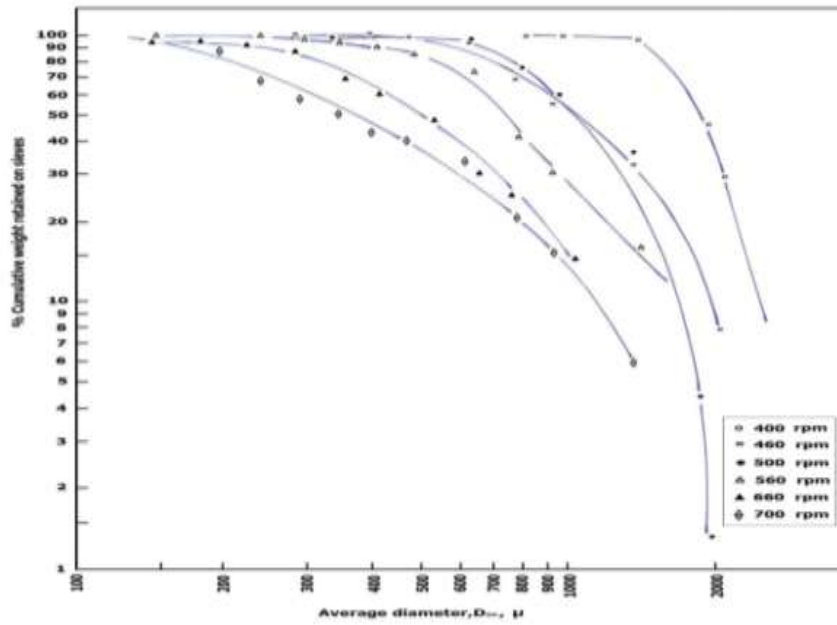


Fig. 2. Cumulative plot of sieve analysis PMMA using 1% starch concentration at variable speed of agitation.

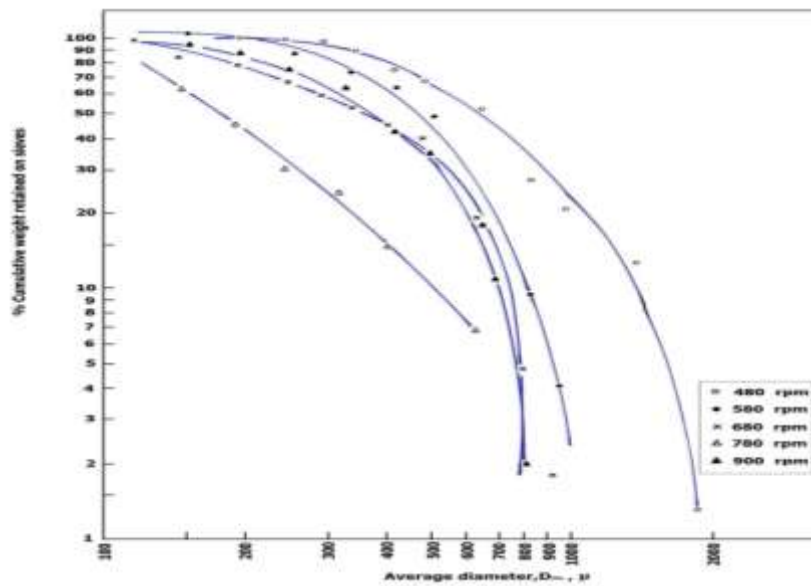


Fig. 3. Cumulative plot of sieve analysis PMMA using 1.5% starch concentration at variable speed of agitation.

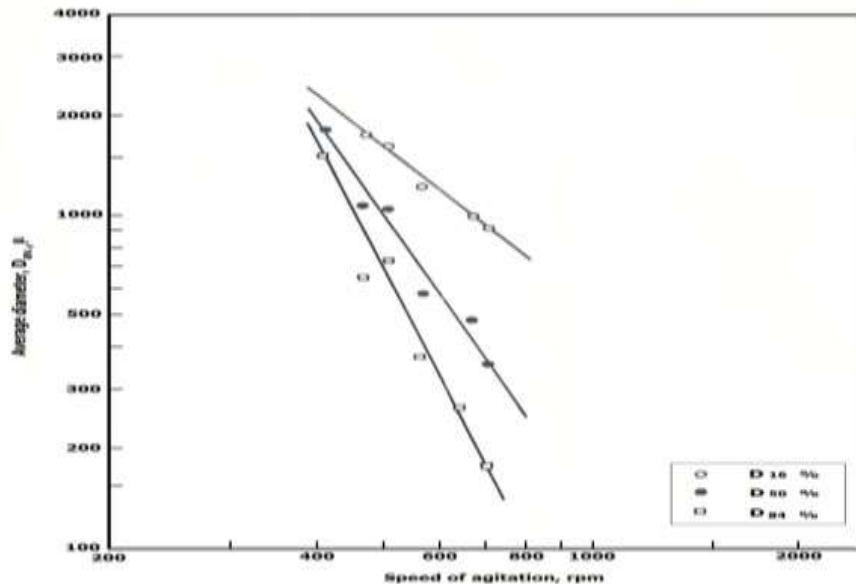
TABLE 6. Variation of $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, dispersion and skew with the speed of agitation (N) at 1% C.

N_{rpm}	$D_{16\%}$	$D_{50\%}$	$D_{84\%}$	Dispersion	Skew
400	1500	1800	2200	350	0.1429
460	640	1050	1720	540	0.2407
500	720	1020	1600	440	0.3182
560	375	570	1215	420	0.5357
660	260	470	980	360	0.4167
700	175	350	900	362.5	0.5172

TABLE 7. Variation of $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, dispersion and skew with the speed of agitation (N) at 1.5 % C.

N_{rpm}	$D_{16\%}$	$D_{50\%}$	$D_{84\%}$	Dispersion	Skew
480	350	600	1160	405	0.3827
580	240	430	840	300	0.3567
680	180	347.5	665	242.5	0.3093
780	105	213	525	210	0.4857
900	185	340	620	217.5	0.2874

Tables 6 and 7 show that as the speed of agitation increases, the particle size decreases. The skew in Table 7 decreases indicating that $D_{84\%}$ approaches $D_{50\%}$. Plotting $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$ against N at 1 and 1.5 % C on log-log paper, the curves in Fig. 4 & 5 are obtained.

**Fig. 4. Log average diameter as function of log speed of agitation at 1% starch concentration .**

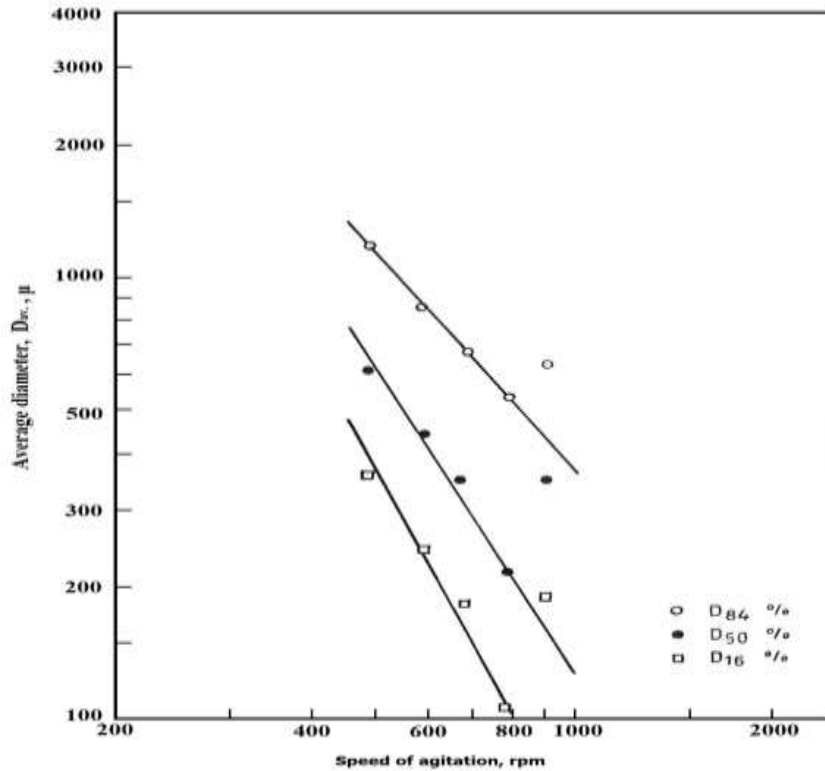


Fig. 5. Log average diameter as function of log speed of agitation at 1.5 % starch concentration .

It is shown that $\log D_{16\%}$, $\log D_{50\%}$ and $\log D_{84\%}$ are inversely proportional to $\log N$ and the relation may be governed by the equation:

$$\log D = A + B \log N$$

where A and B are constants depending on starch concentration. This result contradicts with the previous results on the (¼ t capacity reactor) in which the particles size was directly proportional to the speed of agitation. This contradiction in results was attributed to the prevailing of coalescence of polymer droplets over their breakage in the small reactor especially in the gel stage, this coincides with both Langner *et al.*⁽²²⁾ who worked in a conventional stirred tank reactor at a speed of agitation of $N > 450$ rpm, and Tanaka and Hosogai⁽²³⁾ who worked at a circular loop reactor at a speed of agitation $1300 < N < 1800$ rpm. Scaling up of the reactor allows liter capacity reactor permit water will absorb the heat of polymerization efficiently, accordingly the breakage of the globules prevails their coalescence.

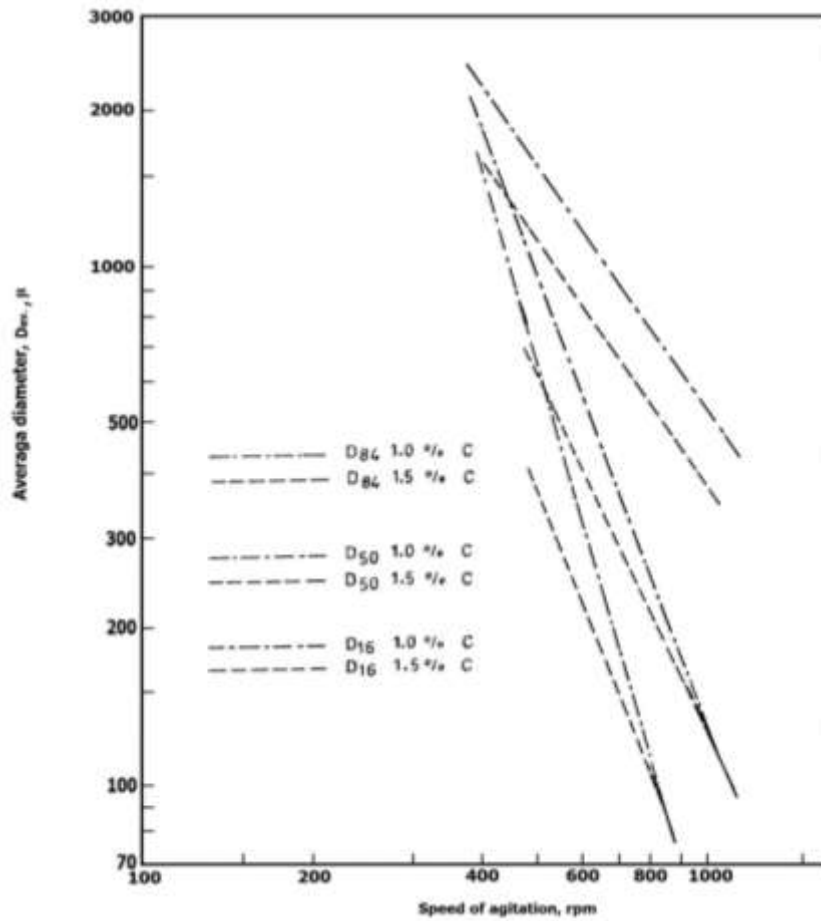


Fig. 6. Log average diameter as function of log speed of agitation at 1.5 % starch concentration .

From Fig. 4 & 5, Fig. 6 is obtained which shows that as the speed of agitation increases, the corresponding curves for $D_{16\%}$, $D_{50\%}$ approach each other till they intersect at a specific point. At this point of intersection the $D_{16\%}$, $D_{50\%}$ has constant values independent of starch concentration.

Effect of the concentration of starch C (0.75-1.75%) at constant speed of agitation N, on the PS and PSD .

Figures 7 & 8 show the cumulative plots retained on sieves against average diameter at 480 and 580 rpm, respectively, at variable starch concentrations.

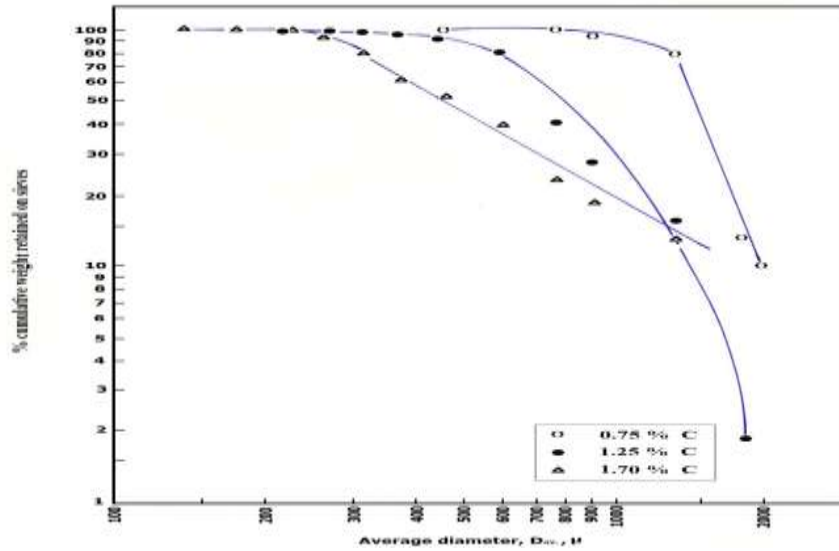


Fig. 7. Cumulative plot of sieve analysis PMMA using 480 rpm speed at variable starch concentration.

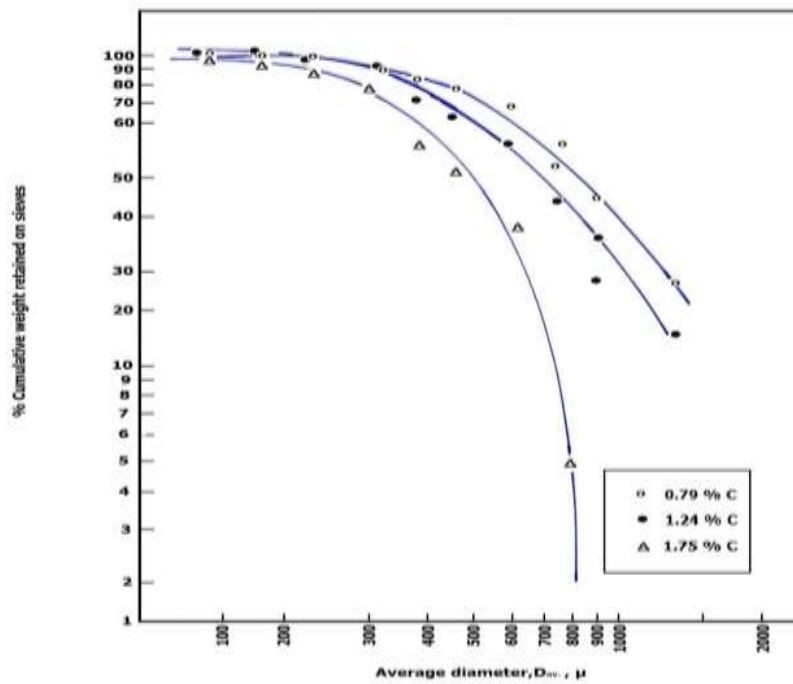


Fig. 8. Cumulative plot of sieve analysis PMMA using 580 rpm at variable starch concentration .

From Fig. 7 and 8, Tables 8 and 9 were obtained which summarize the variation of $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, dispersion and skew with the starch concentration (C) at 480 rpm and 580 rpm, respectively.

TABLE 8. Variation of $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, dispersion and skew with the starch concentration (C) at 480 rpm.

C%	$D_{16\%}$	$D_{50\%}$	$D_{84\%}$	Dispersion	Skew
0.755	1240	1450	1800	280	0.2500
1.00	730	1050	1650	460	0.3043
1.25	550	810	1250	350	0.2575
1.50	350	600	1160	405	0.3827
1.70	295	450	1180	442.5	0.6497
1.75	325	480	1080	377.5	0.5894

TABLE 9. Variation of $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, dispersion and skew with the starch concentration (C) at 580 rpm.

C%	$D_{16\%}$	$D_{50\%}$	$D_{84\%}$	Dispersion	Skew
0.79	385	705	1410	512.5	0.3756
1.00	355	600	1200	422.5	0.4201
1.242	340	585	1150	405	0.4012
1.50	240	435	846	385	0.4545
1.75	265	390	660	197.5	0.3671

Tables 8 & 9 show that on increasing the starch concentration, the particle size decreases. Table 8 shows that both the dispersion and the skew increases with increasing C. Plotting $D_{16\%}$, $D_{50\%}$ and $D_{84\%}$, against C in log-log paper, the curves in Fig. 9 & 10 are obtained.

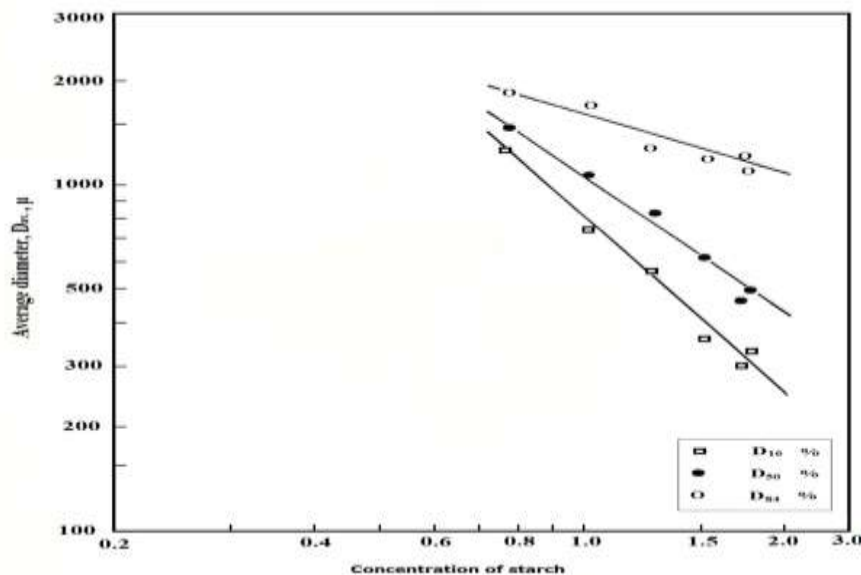


Fig. 9. Log average diameter as function of log concentration of starch at 480 rpm.

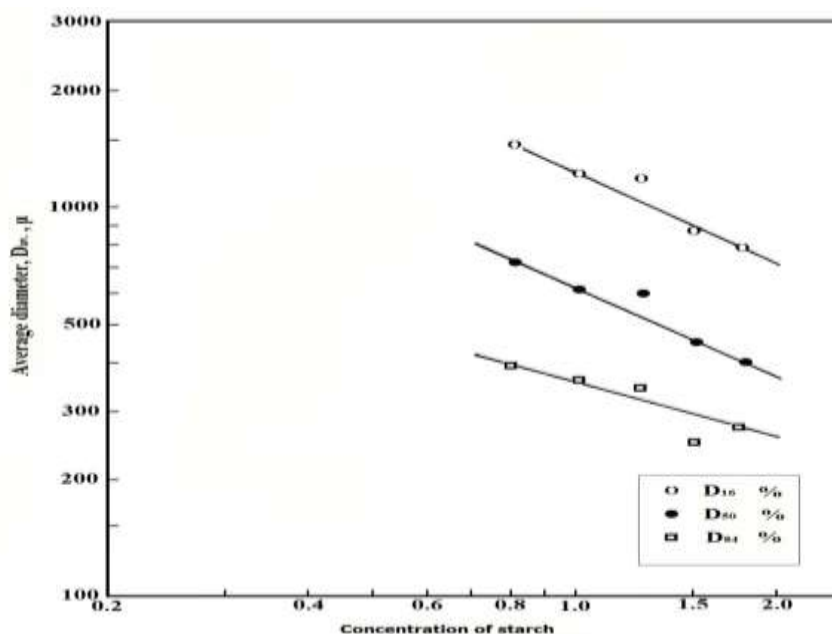


Fig. 9. Log average diameter as function of log concentration of starch at 580 rpm .

It is shown that $\log D_{16\%}$, $\log D_{50\%}$ and $\log D_{84\%}$ are inversely proportional to $\log C$ and the relation may be governed by the equation:

$$\log D = G + H \log C$$

where G and H are constants depending on N .

Graphical analysis data:

It had been concluded that:

$$\log D_{16\%} = A + B \log N$$

$$\log D_{50\%} = A' + B' \log N$$

$$\log D_{84\%} = A'' + B'' \log N$$

where A , A' , A'' , B , B' , and B'' are constants depending on C ,

Also,

$$\log D_{16\%} = G + H \log C$$

$$\log D_{50\%} = G' + H' \log C$$

$$\log D_{84\%} = G'' + H'' \log C$$

where G , G' , G'' , H , H' , and H'' are constants depending on N .

Solution of these equations, by getting values of A , A' , A'' , B , B' , and B'' as function of C or by getting the values of G , G' , G'' , H , H' , and H'' as function of

N, got an equation correlating $\log D_{16\%}$, $\log D_{50\%}$ and $\log D_{84\%}$ to both $\log N$ and $\log C$.

Plotting A, A', A'', B, B', and B'' against $\log C$ as shown in Fig. 11 and 12, it is shown that A, A', and A'' are inversely proportional to $\log C$ and the relation may be governed by the following equations:

$$\begin{aligned} A &= 12.8826 - 17.0664 \log C \\ A' &= 10.7947 - 11.1306 \log C \\ A'' &= 7.5059 - 0.7524 \log C \\ B &= -3.7369 + 5.8120 \log C \\ B' &= -2.8993 + 3.7104 \log C \\ B'' &= -1.5994 \end{aligned}$$

By substitution of the values of A, A', A'', B, B', and B'' in the main equation:

$$\begin{aligned} \log D_{16\%} &= A + B \log N \\ \log D_{50\%} &= A' + B' \log N \\ \log D_{84\%} &= A'' + B'' \log N \end{aligned}$$

The following empirical equations were obtained:

$$\log D_{16\%} = 12.8826 - 17.0664 \log C - 3.7369 \log N + 5.8120 \log N \log C \dots \quad (1)$$

$$\log D_{50\%} = 10.7947 - 11.1306 \log C - 2.8893 \log N + 3.7104 \log N \log C \dots \quad (2)$$

$$\log D_{84\%} = 7.5059 - 0.7524 \log C - 1.5994 \log N \dots \dots \dots \quad (3)$$

Conclusion

In suspension polymerization of methyl methacrylate using starch as protective colloid at 0.75 – 1.75 % concentration and at any speed of agitation between 400 – 900 rpm the relation between the particle size of the product, the concentration of starch and the speed of agitation may be governed by the equations 1, 2, and 3.

Though in the previous article of the same authors⁽²¹⁾ found the particle size was directly proportional to speed of agitation in ¼ liter capacity reactor. The contradiction of the result of this article can be interpreted by the increase of the capacity of the reactors such that the water will absorb the heat of polymerization efficiently, accordingly the breakage of the globules prevails their coalescence.

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دراسة حجم وتوزيع حبيبات البولى ميثيل ميثاكريلات فى البلمرة المائيه المعلقه
لمونومر الميثيل ميثاكريلات فى اثناء تفاعل مصمم بقلاب ذى اربعة حواجز

راجية محمود محسن و يسريه ابو عيانة
قسم البلمرات والمخضبات - المركز القومى للبحوث- القاهرة- مصر.

تعتبر البلمره المائيه المعلقه للمونومرات للحصول على البوليمرات على شكل حبيبات كروييه من أهم أنواع البلمره لتطبيقاتها المختلفه فى شتى المجالات . والتحكم فى العوامل المؤثره فى حجم الحبيبات من أهم الأهداف التى تعمل على البلمره المائيه المعلقه .

وفى هذا البحث تم دراسة عاملين من أهم العوامل المؤثره " سرعة التقليب وقوة تركيز ماده الحافظه والمانع لتجمع الحبيبات" على البلمره المائيه المعلقه لمونومر الميثيل ميثاكريلات فى حجم الحبيبات الكروييه وتوزيعها الحجمى .

وقد وجدنا أن حجم الحبيبات يتناسب عكسيا مع كل من سرعة التقليب وقوة تركيز ماده الحافظه. وقد تم الحصول على معادلات رياضيه تجمع بين حجم الحبيبات والعاملين المؤثرين المشار اليهما أعلاه.