CHAPTER 3

RESULTS AND DISCUSSION

3.1 Preliminary study on conventional emulsion polymerization process

Emulsion polymerization is a free radical polymerization whose kinetics is affected by the presence of small amounts of inhibitors. The presence of inhibitors in a certain level can cause reduction in the rate of reaction. Besides inhibitors or impurities found in chemicals, oxygen is known to act as an inhibitor for many free-radical polymerization systems. In the presence of oxygen, the polymerization rate decreased when the agitation rate increased, namely when the oxygen mass transport rate from the headspace to the aqueous phase increased. Oxygen is most appropriately treated as both water-soluble and monomer-soluble inhibitor [60-61].

The small amount of inhibitor used in technical grade of monomer to stabilize was less critical than oxygen because the inhibitor was completely dissolved in the reaction mixture where the main effect was causing a delay in the start of the polymerization process, unlike the diffusion of the oxygen from the atmosphere to the reaction mixture through the stirring bearing that led to a continuous inhibition period throughout the reaction. An increase in the polymerization rate was observed when the system is purged with nitrogen throughout the reaction to expel out the oxygen. As a result, nitrogen gas was purged throughout the reaction in the emulsion polymerization process.

In this study, styrene and butyl acrylate were used as the main monomers. Polystyrene (PS) has been widely used because of its several advantages; such as low cost, high transparency, high temperature performance, lower permeability, provide higher strength, good chemical resistance, good insulating and processing properties. However, as homopolymer, polystyrene is very brittle, has low toughness and heat resistance. In order to improve the mechanical properties, in particular its impact property, a comonomer of softer properties is often introduced. Acrylic esters such as n-butyl acrylate (nBA) are commercially important in the synthesis because of their optical clarity, mechanical properties, adhesion and chemical stability to apply in paints, adhesives and coatings. It was used as a toughening agent for PS in emulsion copolymerization [63-64]. Hence, styrene and butyl acrylate were chosen to use in our emulsion polymerization process in order to get the T_g in the range of 55 to 65℃ as reported in many patents by using the correct combination ratio of monomers used and low cost factor. Besides, styrene-acrylate resin is widely used in toner application.

Dowfax 2A1 and Ipegal 897 were used as the emulsifiers. The submicron polymer particles are unstable in nature but they can be stabilized by the electrostatic repulsion force provided by anionic surfactant Dowfax 2A1, and steric repulsion force provided by nonionic surfactant Ipegal 897 to overcome the Van der Waals attraction forces between the particles. In emulsion polymerization, anionic surfactants serve as strong particle generators, whereas nonionic surfactants provide the latex with excellent stability toward high electrolyte concentrations, freeze thaw cycling and high shear rates[64]. Polymerization with nonionic surfactant alone resulted in slower rate of polymerization and larger particle size [65]. The amount of coagulum produced by intensive particle coagulation can be greatly reduced when the level of the anionic surfactant increased [63-65].

3.2 Preparation of latex for emulsion-aggregation process

The parameter that was modified in the preparation of latex in emulsion polymerization was the percentage of chain transfer agent (CTA) used in the polymerization. A series of styrene, butyl acrylate and acrylic acid copolymer latex was synthesized with different percentage of 1-dodecanthiol (CTA) while maintaining other parameters unchanged was carried out to study the effect. The raw materials and formulation used in synthesis of these latexes are summarized in Table 2.0, page 38.

The chain transfer agent was used to control the molecular weight distribution of the copolymer. Chain transfer is a polymerization reaction by which the activity of a growing polymer radical is transferred to another molecule as shown in Figure 3.0 below. Generally, a chain transfer reaction does not affect the overall rate of polymerization but able to reduce the molecular weight of the final polymers [37, 66-67].

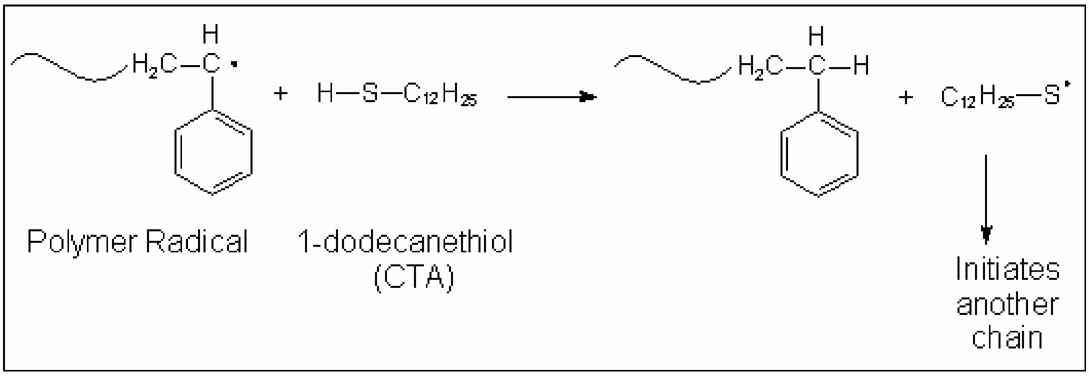

Figure 3.0: The chain transfer reaction of the propagating polymer radical and the CTA

3.3 Characterization of copolymer latexes for emulsion-aggregation process

3.3.1 Total Solid Content (TSC)

Typical determinations of total solid content (TSC) of latexes are shown in Table 3.0. The test was done in triplicates for each sample and the average value was taken. It can be seen that the TSC is in the range of 38.0 to 39.0% which is quite close to the calculated value. The amount of Dowfax 2A1 and Ipegal 897 used in the formulation is 3.9% and 0.1% of the total monomers respectively and potassium persulphate used was 2% weight of total monomers. Based on the total solid content (TSC) of the latex determined by gravimetric method, the conversion of the latexes was 99% as shown in Table 3.1.

Table 3.0: Total Solid Contents (TSC) for latexes

Samples		**Weight of empty Al pan (g)**	**Weight of wet sample (g)**	**Weight of dry sample + Al pan after heating for 3 hours at 110◦C (g)**	**Weight of dry sample (g)**	**% TSC**	**Avg (%)**
E125/07	**i**	0.1560	2.1265	0.9642	0.8082	38.01	38.04
	ii	0.1258	2.4566	1.0598	0.9345	38.04	
	iii	0.1698	2.4560	1.1050	0.9352	38.08	
E127/07	**i**	0.1568	2.3121	1.0358	0.879	38.02	38.05
	ii	0.1287	2.1044	0.9299	0.8012	38.07	
	iii	0.1989	2.1266	1.0081	0.8092	38.05	
E128/07	**i**	0.1668	2.1020	0.9654	0.7986	37.99	38.04
	ii	0.1556	2.1546	0.9754	0.8198	38.05	
	iii	0.1559	2.1578	0.9776	0.8217	38.08	
E129/07	**i**	0.1544	2.1256	0.9624	0.8080	38.01	37.94
	ii	0.1245	2.1545	0.9423	0.8178	37.96	
	iii	0.1642	2.2150	1.0023	0.8381	37.84	
E130/07	**i**	0.1662	2.2145	1.0065	0.8403	37.95	37.98
	ii	0.1241	2.3154	1.0045	0.8804	38.02	
	iii	0.1321	2.3614	1.0285	0.8964	37.96	
E149/08	**I**	0.1452	2.3124	1.0121	0.8669	37.49	37.86
	ii	0.1254	2.2134	0.9645	0.8391	37.91	
	Iii	0.1369	2.3541	1.0356	0.8987	38.18	

Table 3.1: Monomers conversion based on total solid content of the latex determined by gravimetric method

Latex Sample	Calculated TSC at 100% conversion (%)	Experimental TSC (%)	Conversion (%)
E125/07	38.38	38.04	99.11
E127/07	38.38	38.05	99.14
E128/07	38.38	38.04	99.11
E129/07	38.38	37.94	98.85
E130/07	38.38	37.98	98.96
E149/08	38.38	37.86	98.65

3.3.2 Particle Size Distribution (PSD)

Particle sizes distributions of these latexes were measured by CILAS Particle Size Analyzers 1064L. CILAS 1064L is fitted with a 633nm red laser which is able to perform particle size measurement on the various emulsion latexes and raw toners. The samples were first prepared by diluting the latex in approximately 1 to 50 parts of distilled water. The sample was transferred into the sample compartment and allowed the red 633nm laser beam to pass through it. The particle sizes distribution (PSD) of the series of the emulsion latexes are shown in Table 3.2.

Table 3.2: The particle size distribution (PSD) of the latex particle

Samples	**Diameter (μm)**		
	Diameter of particle that is equal to or less than 5% of the mass of particles present, D5	**Average Particle Diameter by mass, D50**	**Diameter of particle that is equal to or less than 95% of the mass of particles present, D95**
E125/07	0.35	0.54	0.82
E127/07	0.17	0.41	0.80
E128/07	0.22	0.57	1.08
E129/07	0.22	0.56	1.07
E130/07	0.23	0.58	1.08
E149/08	0.28	0.59	1.15

Particle size distribution (PSD) is closely related to the product performance in industrial application. In emulsion polymerization processes, the control of the PSD is important due to the direct relation between the PSD and the packing of particles. The control of PSD in emulsion polymerization reactors suffer from the unavailability of frequent online measurement and heterogeneous nature of the process forming a challenging dynamic control problem. There is relatively little literature about the control of the PSD in emulsion polymerization. Study shows that the only factors that can be used to influence the PSD in a controlled fashion are the micellar nucleation, usually for production of a bimodal distribution and particle growth [68].

It is well known that the emulsifier concentration has a strong effect on the average particle diameter (Dv) and the number of polymer particles (N_1) formed in emulsion polymerization. According to the Smith-Ewart micellar theory, the relation between them can be expressed mathematically in correlation 3.0 as:

$$N_1 \; \alpha \, [emulsifier]^{0.6} \quad \textbf{[3.0]}$$

Correlation 3.0 has been proven valid for emulsion polymerization of water-insoluble monomers using anionic surfactant [79]. Another factor which affects Dv and articles, N_1 is the feed monomer composition. In the emulsion copolymerization of styrene and butyl acrylate, the introduction of butyl acrylate monomer causes very slight effect on the final particle size and the polydispersity index. The N_1 decreases slightly with increasing the percentage of butyl acrylate in the feed composition and stated that the number of particles of butyl acrylate is smaller than styrene ones [69].

From the PSD results, the series of latexes showed quite consistent reading of about 0.50 µm which is suitable for emulsion-aggregation process in the following stage as claimed in many patents[30].

3.3.3 Melt Flow Index (MFI)

Melt Flow Index (MFI) of the isolated copolymers from the latexes were determined by Ray-Ran Melt Flow Indexer Serial no RR300/250. The MFI for all the copolymers are summarized in Table 3.3.

Table 3.3: The melt flow index (MFI) of the copolymers

Sample	Time (s)	Sample Weight (g)	MFI (g/10 min)
E125/07	5.1	0.6206	69.01
E127/07	6.4	0.6956	61.21
E128/07	8.9	0.7436	55.13
E129/07	10.6	0.6166	34.94
E130/07	13.2	0.5465	24.84
E149/08	14.3	0.3951	16.58

As expected, with higher percentage of chain transfer agent, the copolymer had lower molecular weight. Thus, there was an increasing trend in the MFI when the percentage of CTA was increased.

3.3.4 Glass Transition Temperature (T_g)

Polystyrene has a T_g of 97.6°C while poly(butyl acrylate) of -45.6°C. The glass transition temperature (T_g) is the temperature at which the amorphous polymer (or the amorphous regions in a partially crystalline polymer) changes from a hard and relatively brittle condition to a rubbery condition. At temperature below the T_g, the polymer segments do not have sufficient energy to rotate or rearrange and thus remain in the rigid and glassy states. As the sample was heated, the volume and energy increased gradually, until it reached the T_g where the mobility of the polymer chains intensified and eventually the polymer could behave more "rubbery" [49,70].

The T_g's of copolymers depend very much on their monomer blocks sequence distribution and has proven to amenable to interpretation based on the measure of copolymer chain flexibility provided by their conformational entropies. To correlate the conformational entropy and the T_g's of copolymers we assume that if the conformational entropy of a copolymer is greater, the copolymer T_g will be lowered and vice-versa [70].

Glass transition temperatures of these copolymers were determined in order to choose the suitable aggregation temperature used in emulsion-aggregation process. The glass transition temperatures of the copolymers are shown in Table 3.4.

Table 3.4: The glass transition temperature (T_g) of the copolymers

Sample ID	Onset (°C)	Midpoint (°C)	End Point (°C)
E125/07	53	57	60
E127/07	54	59	62
E128/07	55	61	64
E129/07	58	62	66
E130/07	57	63	67
E149/08	59	64	69

The midpoint of the glass transition temperature shows an increasing trend when the percentage of chain transfer agent decreases from 2.2% to 1.0%. When the percentage of chain transfer agent is higher, there are more of the shorter polymer chains over the longer polymer chains. As a result, the glass transition temperature decreases significantly because the glass transition temperature depends a lot on the structure of the polymeric chains.

3.3.5 Molecular Weight Distribution

The molecular weight distribution for all these copolymers were measured by GPC system comprising of Waters 1515 Isocratic HPLC Pumps and Waters 2414 Refractive Index Detector in combination of μ-styrogel GPC column. From the measurement done, the molecular weight distribution of all the copolymers is shown in Table 3.5 below.

Table 3.5: The molecular weight distribution of the copolymers

Sample ID	**Molecular Weight Distribution (g/mol)**			
	Mn ($\times 10^4$)	**Mw ($\times 10^4$)**	**Mp ($\times 10^4$)**	**Polydispersity, Mw/Mn**
E125/07	1.08	2.76	1.81	2.56
E127/07	1.06	3.03	1.84	2.86
E128/07	1.18	2.60	2.13	2.19
E129/07	1.23	2.70	2.14	2.18
E130/07	1.36	3.12	2.51	2.29
E149/08	1.87	4.26	3.22	2.28

Molecular weight distribution affects the flow characteristic and mechanical properties. The Mn and Mp showed a decrease when the percentage of the chain transfer agent was increased. The shapes of these curves along with the dependences on the initiator and chain transfer agent concentrations are consistent with the chain stoppage by termination of both the two radical chains and by radical chain to chain transfer agent [71]. Table 3.6 below shows the summary of all the properties on the latexes.

Table 3.6: The properties summary of the latexes and copolymers

Latex Properties		**E125/07**	**E127/07**	**E128/07**	**E129/07**	**E130/07**	**E149/08**	**Unit**
Particle Size Distribution	**D5**	0.35	0.17	0.22	0.22	0.23	0.28	**µm**
	D50	0.54	0.41	0.57	0.56	0.58	0.59	**µm**
	D95	0.82	0.80	1.08	1.07	1.08	1.15	**µm**
	5 µm	100.00	100.00	100.00	100.00	100.00	100.00	%
	24 µm	100.00	100.00	100.00	100.00	100.00	100.00	%
Total Solid Content		38.40	38.61	38.43	38.59	38.36	37.86	%
Copolymer Properties								
Melt Flow Index at 130°C		69.01	61.21	55.13	34.90	24.84	16.58	**g/ 10 min**
Glass Transition Temperature (Midpoint)		57	59	61	62	63	64	**°C**
Molecular Weight Distribution	**M_n (10^4)**	1.07	1.16	1.27	1.42	1.49	1.87	**g/mol**
	M_w (10^4)	3.24	3.07	3.83	3.37	3.12	4.26	**g/mol**
	M_p (10^4)	1.92	2.25	2.25	2.25	2.66	3.22	**g/mol**
	Polydispersity, M_w/M_n	3.02	2.65	2.39	2.39	2.12	2.28	**-**

3.4 Emulsion-aggregation process (EA Process) by varying different parameters

Many series of experiments were carried out, selected significant ones were chosen for discussion here as shown in Table 2.2, page 52. Each series of experiments was carried by varying only one of the parameters as shown in Table 2.2 to study the effect of each dependency. After completing all the series, the finished toner is produced based on the best parameters obtained from all series.

All the experiments were carried out using latex E125/07 because with reference to Table 3.6, copolymer latex E125/07 has the nearest glass transition temperature with what most of the patents claimed in the range of 56-57°C. Hence, latex E125/07 was chosen for all the experiments except for series VIII that study the effect on latexes with different percentage of CTA on emulsion-aggregation process. All the raw toners were washed and dried thoroughly before characterization was carried out.

3.4.1 Effect of amount of PAC

The physical appearance of the PAC is a yellowish solution. The exact amount of raw materials used for Series I are shown in the Table 3.7. The calculation of the amount used is based on 60.0g final raw toner produced based on the dry weight of each raw materials as shown in Chapter 2.

Table 3.7: Raw materials composition in weight (g) used for series I

Sample ID	**Latex E125/07 (g)**	**Pigment Dispersion (g) (TSC = 20%)**	**Wax Dispersion (g) (TSC = 35%)**	**Distilled Water (g)**	**PAC (g) (10% w/w solution)**
ME12/07	138.01	15.00	13.70	280.35	**0.90**
ME17/07	138.01	15.00	13.70	280.35	**1.80**
ME14/07	138.01	15.00	13.70	280.35	**2.00**
ME19/07	138.01	15.00	13.70	280.35	**2.50**
ME21/07	138.01	15.00	13.70	280.35	**3.00**

With the reference to Table 3.8, the particle size of the raw toner was seen to have increased as the amount of the PAC was increased in the aggregation process. When the PAC was insufficient (case ME12/07), besides having very small particles, tiny hard "stone" like coarse particles were formed instead of the fine raw toner. The T_g of the raw toner decreased slightly as to compare to the T_g of the copolymer latex itself at 60.14°C before emulsion-aggregation process because of the incorporated wax. Incorporation of wax has shifted the T_g of the raw toner to slightly lower in the range of 50-53℃. Besides, the melt flow index of the raw toner too has been shifted to slightly higher compare to the copolymer latex because of the presence of the wax. The molecular weight distribution of the raw toners does not change much as compare to the molecular weight distribution of the copolymer latex because EA is not a polymerization process that would build up the molecular weight distribution. The other properties did not show any significant differences and quite consistent. As for the fusing, all the cases were able to fuse well where many spherical particles were seen as shown in Figure 3.1.

According to published references [35, 38, 72], the amount of coagulant initially added must be sufficient to impart a desired viscosity to the system to avoid the formation of excessive amount of fines which may occur if the viscosity is too low and insufficient homogenization being achieved which may occur if the viscosity is too high when too much coagulant is added. Hence, the amount of PAC used can neither be too little nor too much to avoid formation of fines particles as well as growth of oversize particles.

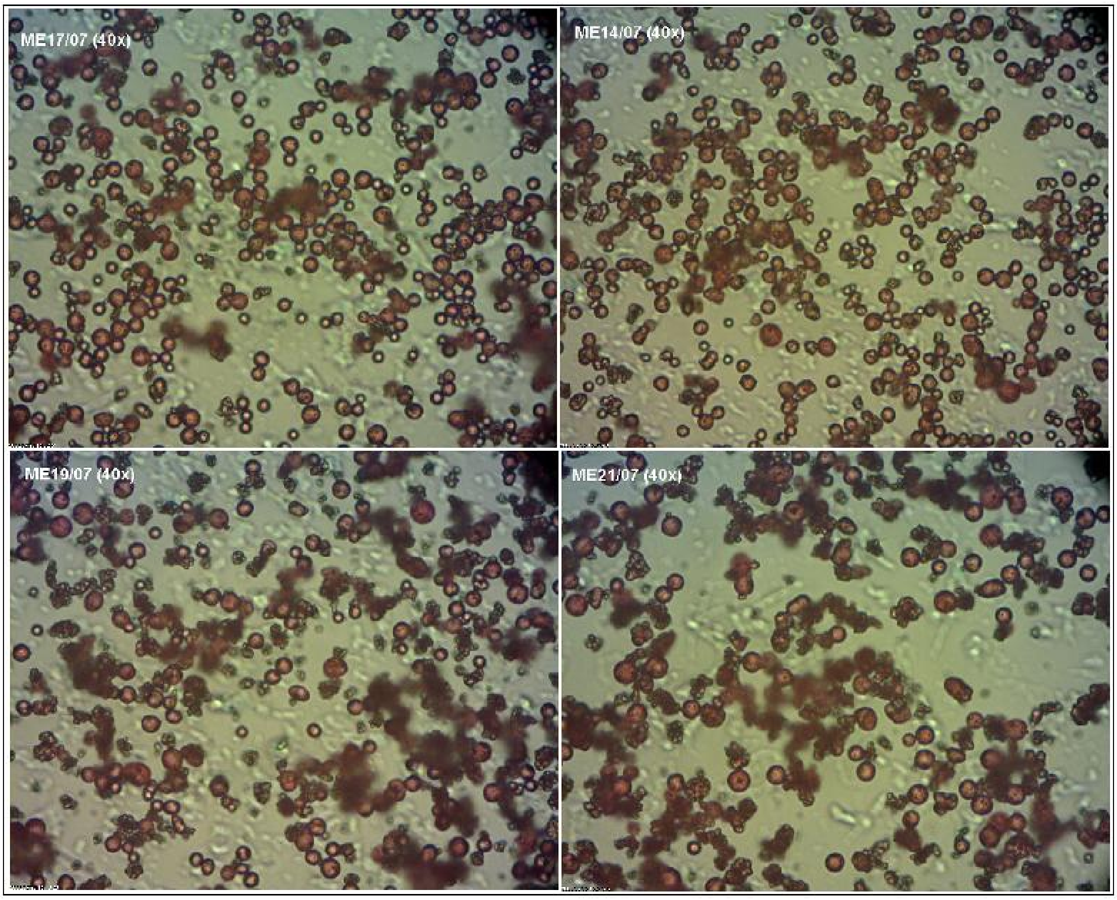

Figure 3.1: The microscopic picture of toner particles series I

Table 3.8: The properties of raw toners series I

Properties		**Average Value Measured**				**Unit**
		ME17/07	**ME14/07**	**ME19/07**	**ME21/07**	
Particle Size Distribution (PSD)	**D5**	0.78	0.74	0.74	0.80	**µm**
	D50	**5.73**	**5.46**	**5.64**	**6.11**	**µm**
	D95	14.42	16.13	14.48	15.61	**µm**
	5 µm	39.89	42.07	40.13	35.32	**%**
	24 µm	99.34	98.83	99.21	98.97	**%**
Sharpness Index		18.49	21.80	19.57	19.51	**-**
Tribocharge		-7.5	-6.30	-6.54	-7.20	**µC/g**
Relative Dielectric Constant.		2.21	2.13	2.14	2.22	**-**
Dissipation Factor		121	102	119	120	**-**
Melt Flow Index at 130°C		76.27	73.24	74.21	73.13	**g/10 min**
Glass Transition Temperature, T_g		51	51	51	53	**°C**
Wax Melting Point, T_p		76	76	76	76	**°C**
Wax Content		6.86	6.93	6.93	6.82	**%**
Molecular Weight Distribution	**M_n (10^4)**	1.11	1.44	1.20	1.26	**g/mol**
	M_w (10^4)	2.40	2.73	2.81	2.91	**g/mol**
	M_p (10^4)	1.73	2.01	1.90	2.01	**g/mol**
	M_w/M_n	2.15	1.90	2.34	2.31	**-**

3.4.2 Effect of aggregation pH

All the other parameters were maintained as shown in Table 3.9 except the aggregation pH was adjusted to pH 1.0, 2.5 and 3.0 using 0.2M nitric acid and 4% sodium hydroxide before aggregation process.

Table 3.9: Raw materials composition in weight (g) used for series II

Sample ID	**Aggregation pH**	**Latex E125/07 (g)**	**Pigment Dispersion (g) (TSC = 20%)**	**Wax Dispersion (g) (TSC = 35%)**	**Distilled Water (g)**	**PAC (g) (10% w/w solution)**
ME02/07	**1.0**	138.01	15.00	13.70	280.35	2.0
ME01/07	**2.5**	138.01	15.00	13.70	280.35	2.0
ME03/07	**3**	138.01	15.00	13.70	280.35	2.0

With the reference to Table 3.10, the particle size of case ME02/07 using pH 1 has relatively smaller particles as to compare to the higher pH. Aggregation process depends very much on the collision frequency between the particles and the collision efficiency which determines whether having collided to remain aggregated. It is suggested that because the initial particle size is much smaller than the Kolmogoroff eddy size, the collision frequency of the particles at this stage is caused by velocity gradients associated with eddies in the viscous sub range of turbulence and by the Brownian motion. At the same time, the reduction of inter particle repulsive forces due to the change of pH enhances the collision frequency. Anyhow, the reduction of pH has the limitation to pH 2.0 and not lesser [63, 73-74].

Besides, PAC is an inorganic chloride salt that required acidic ambient to be reactive but lowering the pH below 2.5 slow down the formation of the particle size. This indicates that the optimum pH for the PAC to aggregate is pH 2.5 to 3.0. The other properties of the raw toners did not show any significant differences. In terms of fusing, all the three cases fused with mixture of spherical and 'potatoes' shapes. The aggregation pH does not affect the fusing efficiency that much. Figure 3.2 shows the microscopic pictures for series II

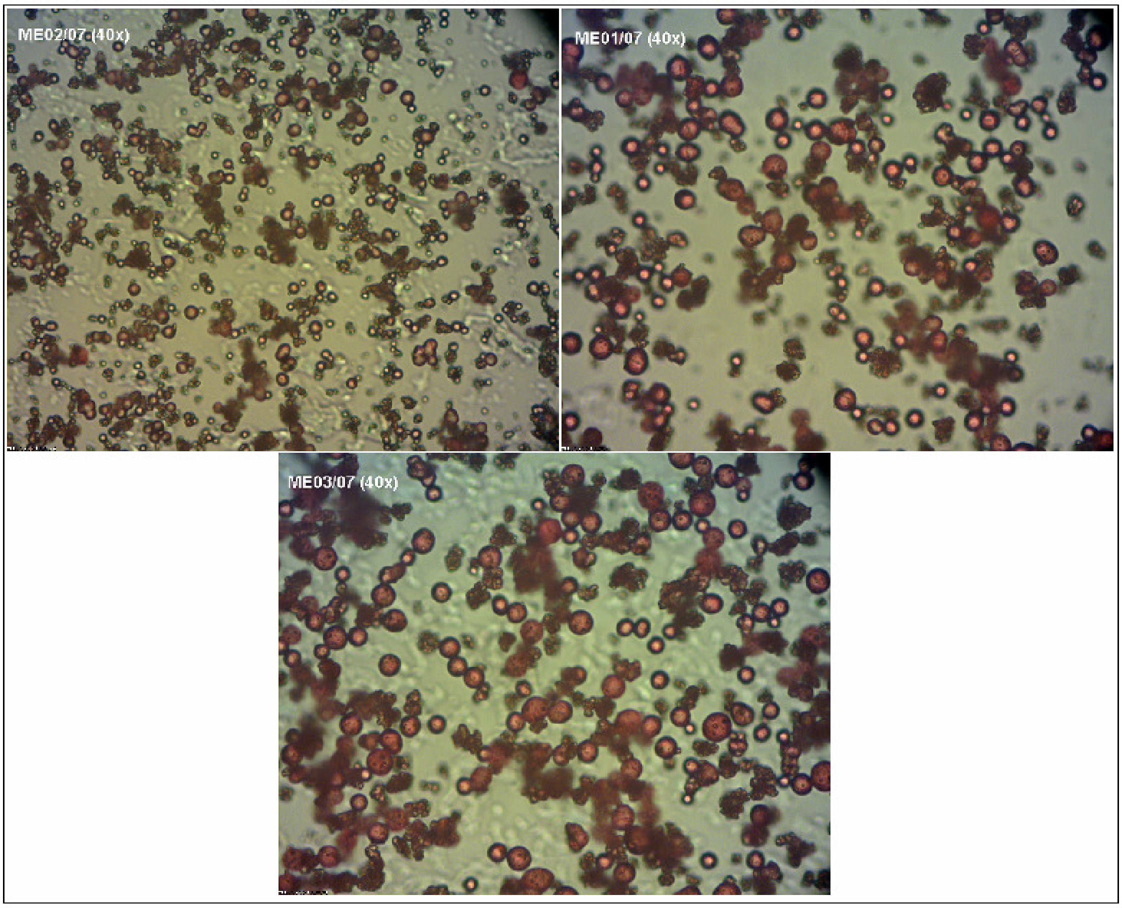

Figure 3.2: The microscopic pictures for toner particles series II

Table 3.10: The properties summary of raw toners series II

Properties		**Average Value Measured**			**Units**
		ME02/07	**ME01/07**	**ME03/07**	
Particle Size Distribution (PSD)	**D5**	0.75	0.85	0.78	**µm**
	D50	**4.40**	**6.55**	**6.80**	**µm**
	D95	13.06	13.95	14.13	**µm**
	5 µm	56.72	29.32	27.15	%
	24 µm	99.87	99.87	99.88	%
Sharpness Index		17.42	16.41	18.12	**-**
Tribocharge		-6.5	-6.0	-7.40	**µC/g**
Relative Dielectric Constant.		2.12	2.16	2.20	**-**
Dissipation Factor		121.2	129.8	122.4	**-**
Melt Flow Index at 130°C		71.20	75.69	73.10	**g/10 min**
Glass Transition Temperature, T_g		53	52	53	**°C**
Wax Melting Point, T_p		76	76	76	**°C**
Wax Content		6.97	6.69	6.99	%
Molecular Weight Distribution	**M_n (10^4)**	1.26	1.35	1.30	**g/mol**
	M_w (10^4)	2.66	2.99	2.99	**g/mol**
	M_p (10^4)	1.95	2.02	1.94	**g/mol**
	M_w/M_n	2.12	2.21	2.30	**-**

3.4.3 Effect of aggregation time

All the other parameters were maintained constant as shown in the Table 3.11 below except for the aggregation time were prolonged from 2 to 6 hours.

Table 3.11: Raw materials composition in weight (g) used for series III

Sample ID	Aggregation Time (hours)	Latex E125/07 (g)	Pigment Dispersion (g) (TSC = 20%)	Wax Dispersion (g) (TSC = 35%)	Distilled Water (g)	PAC (g) (10% w/w solution)
CE20/07	**2**	138.01	15.00	13.70	280.35	2.0
CE33/07	**3**	138.01	15.00	13.70	280.35	2.0
CE34/07	**4**	130.01	15.00	13.70	280.35	2.0
ME05/07	**5**	138.01	15.00	13.70	280.35	2.0
ME08/07	**6**	138.01	15.00	13.70	280.35	2.0

From the results obtained shown in Table 3.12, in the emulsion-aggregation process, there are no significant differences in PSD when the aggregation hours were prolonged. According to references [73-74], the aggregation is normally considered to depend on the collision frequency between particles and the collision efficiency which determines whether having collided they remain aggregated. The collision frequency between particles is very much depending on the initial pH that can destabilize the latex and allow aggregation to occur rather than the aggregation duration. Figure 3.3 shows the microscopic picture of all the cases.

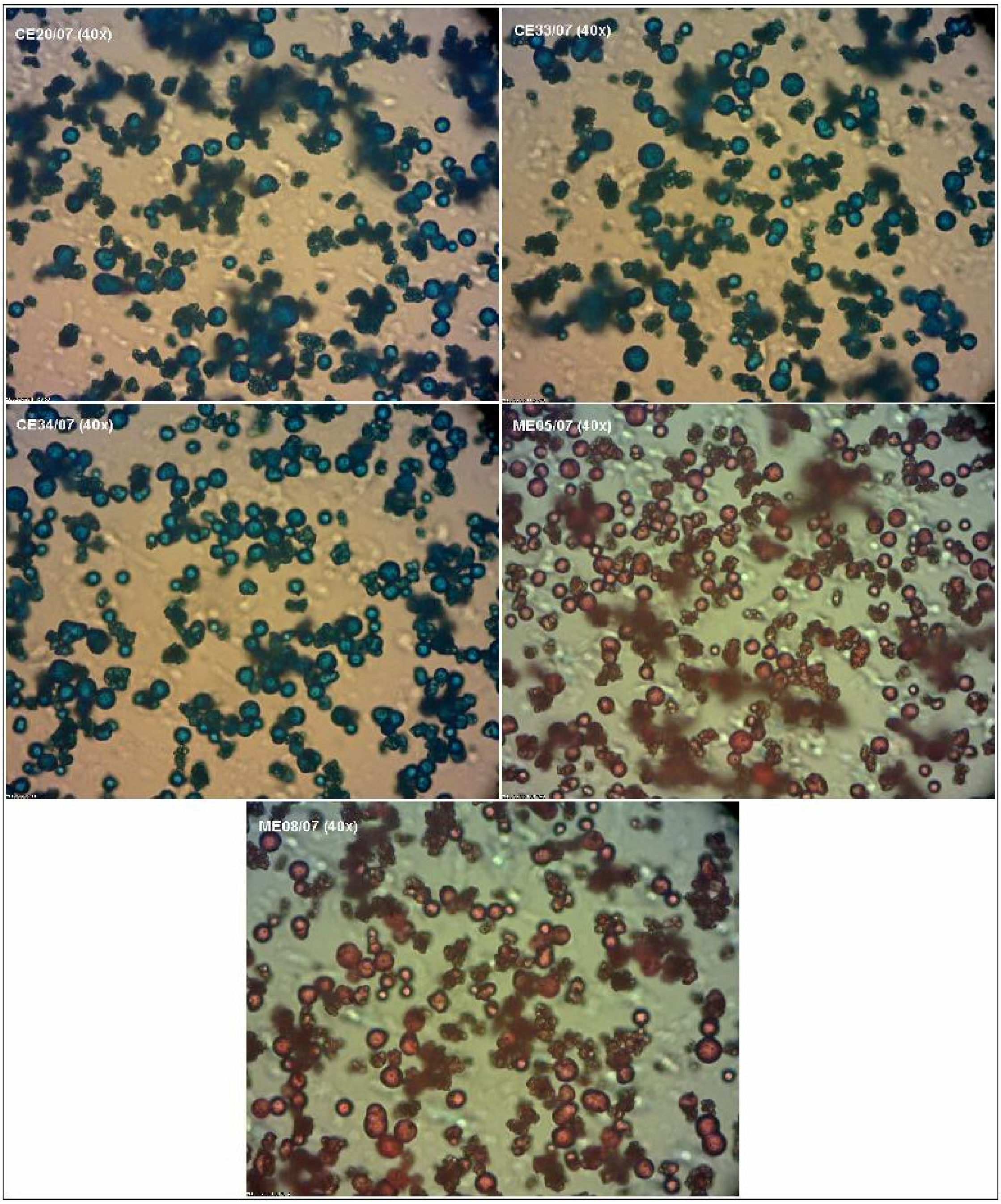

Figure 3.3: The microscopic pictures of toner particles series III

Table 3.12: The properties summary of raw toners series III

Properties		**Average Value Measured**					**Unit**
		CE20/07	**CE33/07**	**CE34/07**	**ME05/07**	**ME08/07**	
Particle Size Distribution (PSD)	**D5**	0.82	0.90	0.91	0.79	0.93	**µm**
	D50	**7.48**	**7.07**	**6.56**	**7.23**	**8.17**	**µm**
	D95	12.82	14.43	15.65	13.27	15.36	**µm**
	5 µm	20.94	25.02	29.72	21.27	15.04	**%**
	24 µm	100.00	100.00	100.00	100.00	99.89	**%**
Sharpness Index		15.63	16.03	17.20	16.80	16.52	**-**
Tribocharge		-6.8	-6.5	-7.5	-7.2	-7.0	**µC/g**
Relative Dielectric Constant.		2.31	2.15	2.07	2.26	2.16	**-**
Dissipation Factor		124.8	119.6	123.8	124.2	127.4	**-**
Melt Flow Index at 130°C		76.80	73.51	72.12	73.10	61.59	**g/ 10min**
Glass Transition Temperature, T_g		53	52	52	51	51	**°C**
Wax Melting Point, T_p		75.88	75.73	75.87	75.56	75.56	**°C**
Wax Content		7.05	6.64	6.99	6.71	6.71	**%**
Molecular Weight Distribution	**M_n (10^4)**	1.35	1.36	1.30	1.44	1.53	**g/mol**
	M_w (10^4)	2.77	3.33	4.91	4.54	3.07	**g/mol**
	M_p (10^4)	2.09	2.02	1.99	2.06	2.22	**g/mol**
	M_w/M_n	2.05	2.45	3.77	3.16	2.01	**-**

3.4.4 Effect of coalescences pH

All the other parameters were maintained as shown in the Table 3.13 below except the coalescences pH was adjusted to pH 4.0, 5.0 and 6.0 at coalescence stage using 0.2M nitric acid and 4% sodium hydroxide.

Table 3.13: Raw materials composition in weight (g) used for series IV

Sample ID	**Coalescence pH**	**Latex E125/07 (g)**	**Pigment Dispersion (g) (TSC = 20%)**	**Wax Dispersion (g) (TSC = 35%)**	**Distilled Water (g)**	**PAC (g) (10% w/w solution)**
CE21/07	**4**	138.01	15.00	13.70	280.35	2.0
CE19/07	**5**	138.01	15.00	13.70	280.35	2.0
CE14/07	**6**	138.01	15.00	13.70	280.35	2.0

With the reference to Table 3.14, the particle size has increased as the coalescences pH was reduced. The pH was adjusted to near neutral to stabilize the aggregates, the temperature was raised well above T_g so that the aggregates internally coalesced to give essentially near-spherical particles. At pH 4 and pH 5, the acidic ambient allowed the excess polyaluminum chloride (PAC) to be active and readily aggregated the other additives such as wax dispersion, pigment dispersion and charge control agent together on the resin particles becoming bigger particles [73-74]. The fusing of the particles is shown in Figure 3.4. The fusing of the particles shown that at pH 6, the particles can fuse the best and hence, pH 6 is adopted in the later section.

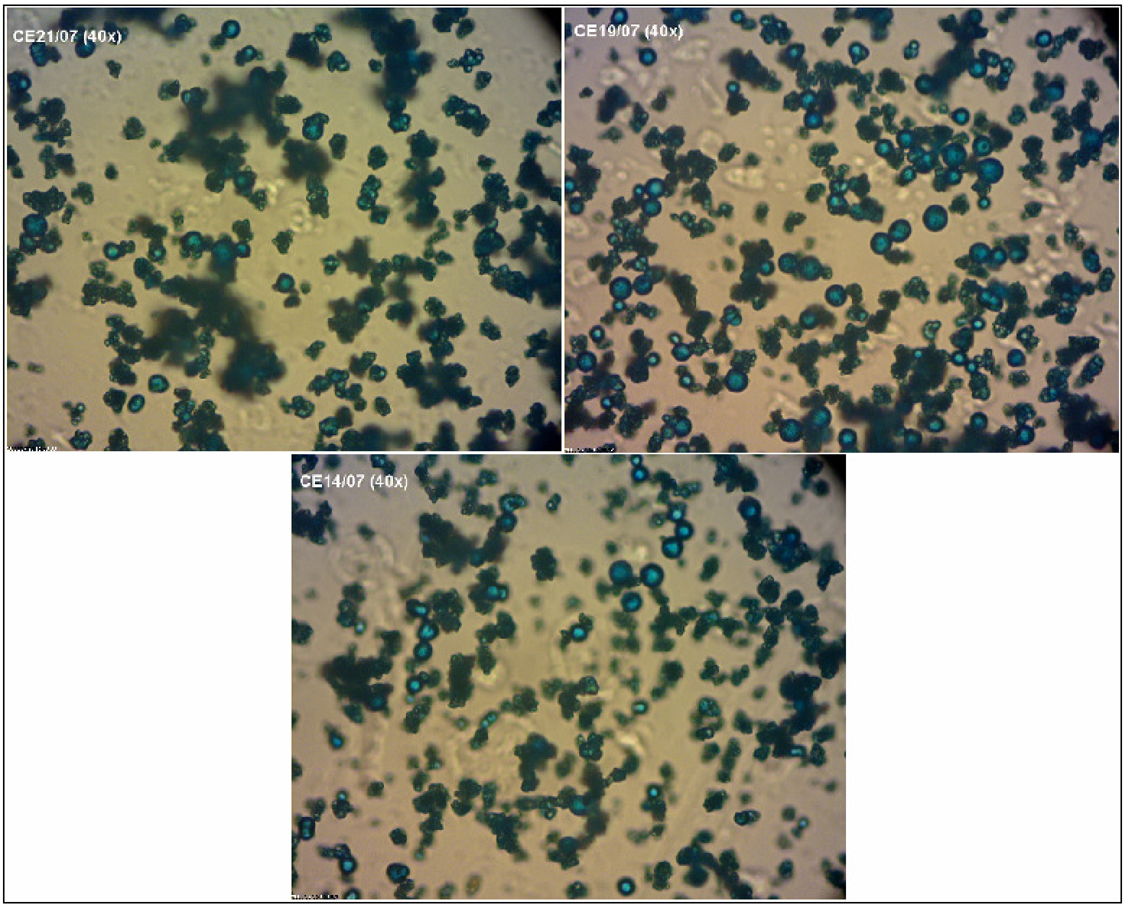

Figure 3.4: The microscopic pictures of toner particles series IV

Table 3.14: The properties summary of raw toners series IV

Properties		**Average Value Measured**			**Units**
		CE21/07	**CE19/07**	**CE14/07**	
Particle Size Distribution (PSD)	**D5**	0.93	0.92	0.92	**µm**
	D50	**7.49**	**7.01**	**6.65**	**µm**
	D95	17.50	12.78	13.27	**µm**
	5 µm	23.51	26.13	29.53	%
	24 µm	99.48	100.00	100.00	%
Sharpness Index		18.81	13.89	14.42	**-**
Tribocharge		-8.00	-7.00	-7.50	**µC/g**
Relative Dielectric Constant.		2.33	2.35	2.21	**-**
Dissipation Factor		126.2	121.0	125.1	**-**
Melt Flow Index at 130°C		60.21	63.45	59.64	**g/ 10 min**
Glass Transition Temperature, T_g		53	54	53	**°C**
Wax Melting Point, T_p		76	76	76	**°C**
Wax Content		6.61	6.91	6.62	%
Molecular Weight Distribution (MWD)	**M_n (10^4)**	1.57	1.53	1.35	**g/mol**
	M_w (10^4)	4.52	5.22	3.84	**g/mol**
	M_p (10^4)	2.27	2.31	2.19	**g/mol**
	M_w/M_n	2.87	3.42	2.85	**-**

3.4.5 Effect of coalescence temperature

All the raw materials were maintained as shown in the Table 3.15 except for the coalescence temperature was adjusted to **(80, 90, 95°C)** before coalescence stage started.

Table 3.15: Raw materials composition in weight (g) used for series V

Sample ID	**Coalescence Temperature (°C)**	**E125/07 (g)**	**Pigment Dispersion (g) (TSC = 20%)**	**Wax Dispersion (g) (TSC = 35%)**	**Distilled Water (g)**	**PAC (g) (10% w/w solution)**
CE15/07	**80**	138.01	15.00	13.70	280.35	2.0
CE09/07	**90**	138.01	15.00	13.70	280.35	2.0
CE11/07	**95**	138.01	15.00	13.70	280.35	2.0

With the reference to Table 3.16, the particle size increased as the coalescences temperature reduced. The pH was adjusted to near neutral to about pH 6-7 to stabilize the aggregates and the temperature was raised well above the copolymer resin T_g so that the aggregates internally coalescence to give essentially near-spherical particles which statically bounded particles aggregated particles comprised of resin, pigment and wax [37]. Hence, when the temperature is insufficient high for the particles to coalesce well, the additives that are bounded to the copolymer resin in the aggregation stage will loose out and resulted smaller particles unlike particles with sufficient temperature to coalesce well and give a bigger particles. The fusing of the particles shown in Figure 3.5 also proved that higher coalescence temperature will give better fusing with many spherical particles as compared to lower temperature at 80 and 90°C with many non-fused particles.

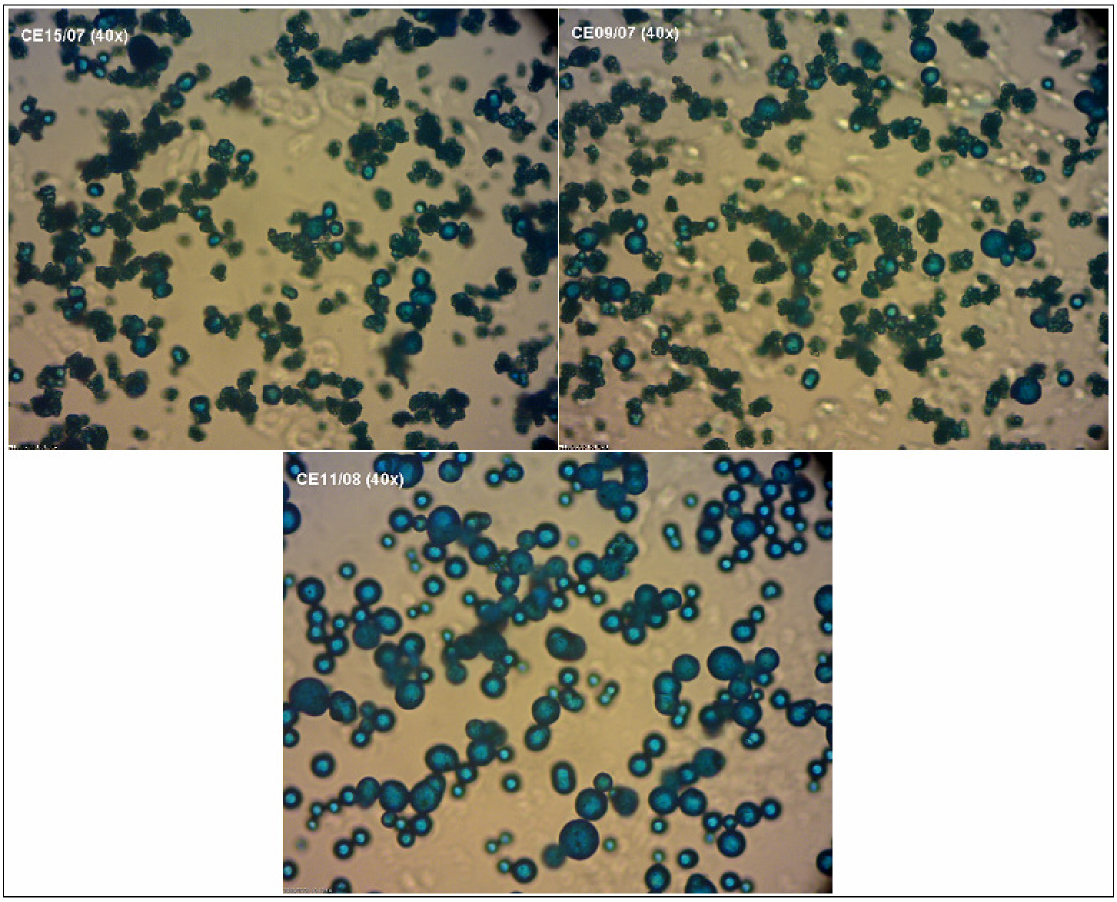

Figure 3.5: The microscopic pictures for toner particles series V

Table 3.16: The properties summary of raw toners series V

Properties		**Average Value Measured**			**Units**
		CE15/07	**CE09/07**	**CE11/07**	
Particle Size Distribution (PSD)	**D5**	0.92	0.89	0.90	**µm**
	D50	**6.55**	**7.19**	**7.83**	**µm**
	D95	11.71	13.18	17.25	**µm**
	5 µm	30.02	25.33	23.87	%
	24 µm	100.00	100.00	99.65	%
Sharpness Index		12.73	14.82	19.17	**-**
Tribocharge		-6.63	-6.50	-7.5	**µC/g**
Relative Dielectric Constant.		2.17	2.14	2.24	**-**
Dissipation Factor		111.2	124.0	120.3	**-**
Melt Flow Index at 130°C		72.2	70.24	69.89	**g/10 min**
Glass Transition Temperature, T_g		53	53	54	**°C**
Wax Melting Point, T_p		76	76	76	**°C**
Wax Content		6.87	6.64	6.79	%
Molecular Weight Distribution (MWD)	**M_n (10^4)**	1.49	1.36	1.45	**g/mol**
	M_w (10^4)	3.92	3.49	5.38	**g/mol**
	M_p (10^4)	2.25	2.14	2.20	**g/mol**
	M_w/M_n	2.57	2.58	3.72	**-**

3.4.6 Effect of incorporated of PAC with second coagulant, benzylkonium chloride

All the other parameters were maintained as shown in the Table 3.17 below except an addition cationic coagulant; benzylkonium chloride was added in different amount.

Table 3.17: Raw materials composition in weight (g) used for series VI

Sample ID	E125/07 (g)	Pigment Dispersion (g) (TSC = 20%)	Wax Dispersion (g) (TSC = 35%)	Distilled Water (g)	PAC (g) (10% w/w solution)	Benzylkonium chloride (g)
YE01/07	138.01	15.00	13.70	280.35	2.00	**0.00**
YE14/07	138.01	15.00	13.70	280.35	2.00	**0.05**
YE16/07	138.01	15.00	13.70	280.35	2.00	**0.07**
YE13/07	138.01	15.00	13.70	280.35	2.00	**0.10**
YE15/07	138.01	15.00	13.70	280.35	2.00	**0.15**

With reference to results in Table 3.18 below showed a drastic increased in the particle size as the second cationic coagulant, benzylkonium chloride was introduced. This product is a nitrogenous cationic surface-acting agent belonging to the quaternary ammonium group. It is mainly used as a cationic surfactant in the toner industry. The latex itself contains the anionic surfactant, Dowfax 2A1, and thus with the addition of the cationic surfactant, benzalkonium chloride of the opposite charge polarity, the aggregation can be accomplished with shorter duration for the same desired particle sizes [39].

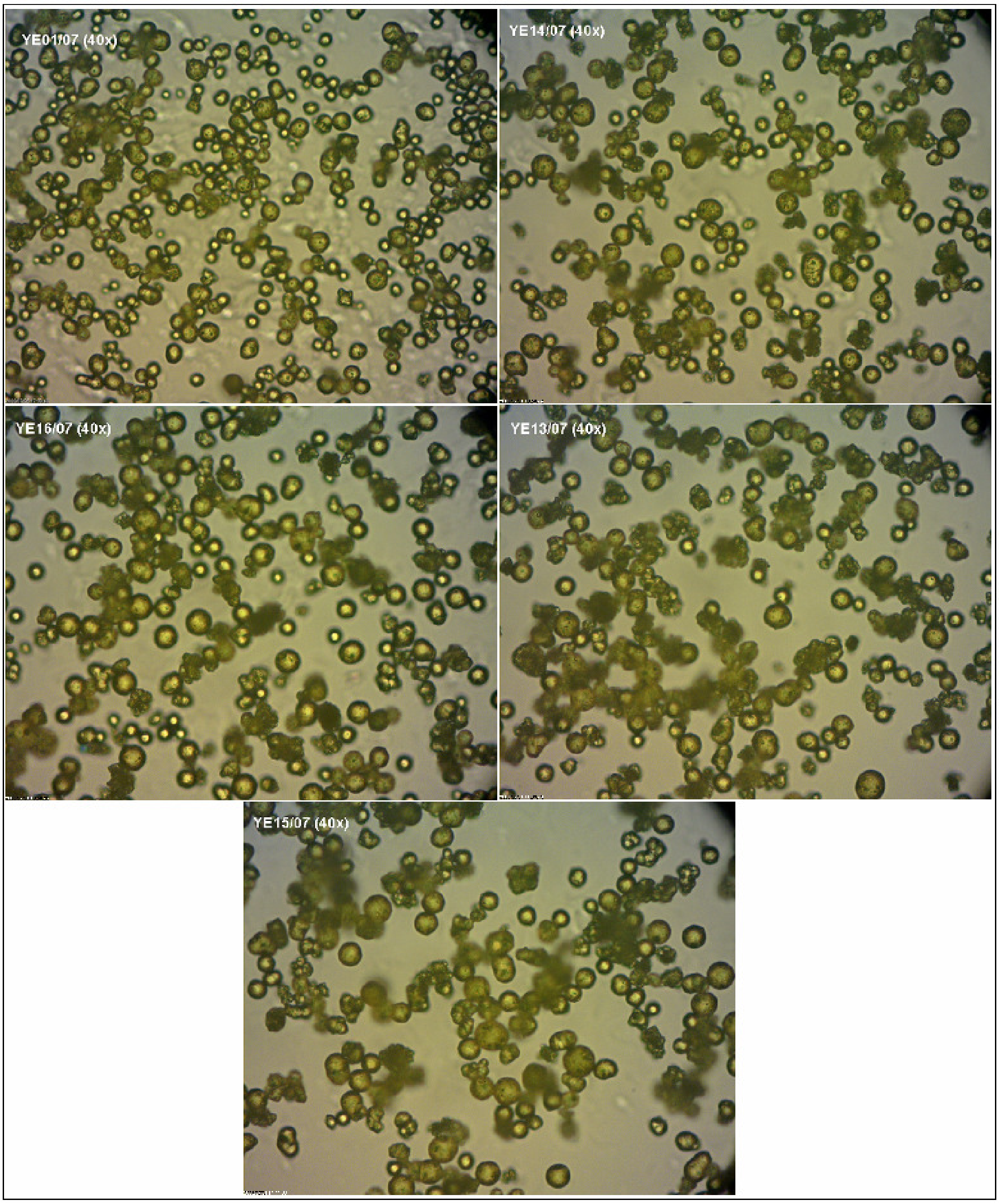

Figure 3.6: Microscopic pictures of toner particles series VI

Table 3.18: The properties summary of raw toners series VI

Properties		**Average Value Measured**					**Unit**
		YE01/07	**YE14/07**	**YE16/07**	**YE13/07**	**YE15/07**	
Particle Size Distribution (PSD)	**D5**	0.79	0.81	0.90	0.84	0.94	**µm**
	D50	**6.28**	**6.88**	**7.94**	**7.52**	**9.33**	**µm**
	D95	15.11	14.28	15.41	14.62	16.97	**µm**
	5 µm	29.19	22.79	15.25	21.86	12.70	**%**
	24 µm	99.69	99.80	99.76	99.86	99.77	**%**
Sharpness Index		19.13	17.63	17.12	17.31	18.05	**-**
Tribocharge		-7.0	-6.0	-7.5	-7.0	-7.2	**µC/g**
Relative Dielectric Constant.		2.20	2.19	2.390	2.35	2.14	**-**
Dissipation Factor		120.5	123.0	128.2	122.4	121.4	**-**
Melt Flow Index at 130°C		75.22	73.25	70.25	71.59	73.02	**g/10 min**
Glass Transition Temperature, T_g		52	51	52	53	50	**°C**
Wax Melting Point, T_p		76	76	76	76	76	**°C**
Wax Content		6.89	6.62	6.95	6.69	6.57	**%**
Molecular Weight Distribution (MWD)	**M_n (10^4)**	0.94	1.13	1.28	1.30	1.29	**g/mol**
	M_w (10^4)	2.83	2.45	2.77	2.67	2.82	**g/mol**
	M_p (10^4)	2.11	1.76	2.01	2.08	2.04	**g/mol**
	M_w/M_n	3.0	2.17	2.16	2.06	2.19	**-**

3.4.7 Effect of percentage of wax

All the other parameters were maintained as shown in the Table 3.19 below except the wax dispersion was added in different percentage calculated based on 60.0g dry weight of raw toner as shown in calculation method in Chapter 2, section 2.4, pg 50.

Table 3.19: Raw materials composition in weight (g) used for series VII

Sample ID	**E125/07 (g)**	**Pigment Dispersion (g) (TSC = 20%)**	**Wax Dispersion (TSC = 35%)**		**Distilled Water (g)**	**PAC (g) (10% w/w solution)**
			% of 60g raw toner dry weight	**(g)**		
YE02/07	138.01	15.00	**6**	13.70	280.35	2.00
YE09/07	138.01	15.00	**7**	13.70	280.35	2.00
YE01/07	138.01	15.00	**8**	13.70	280.35	2.00

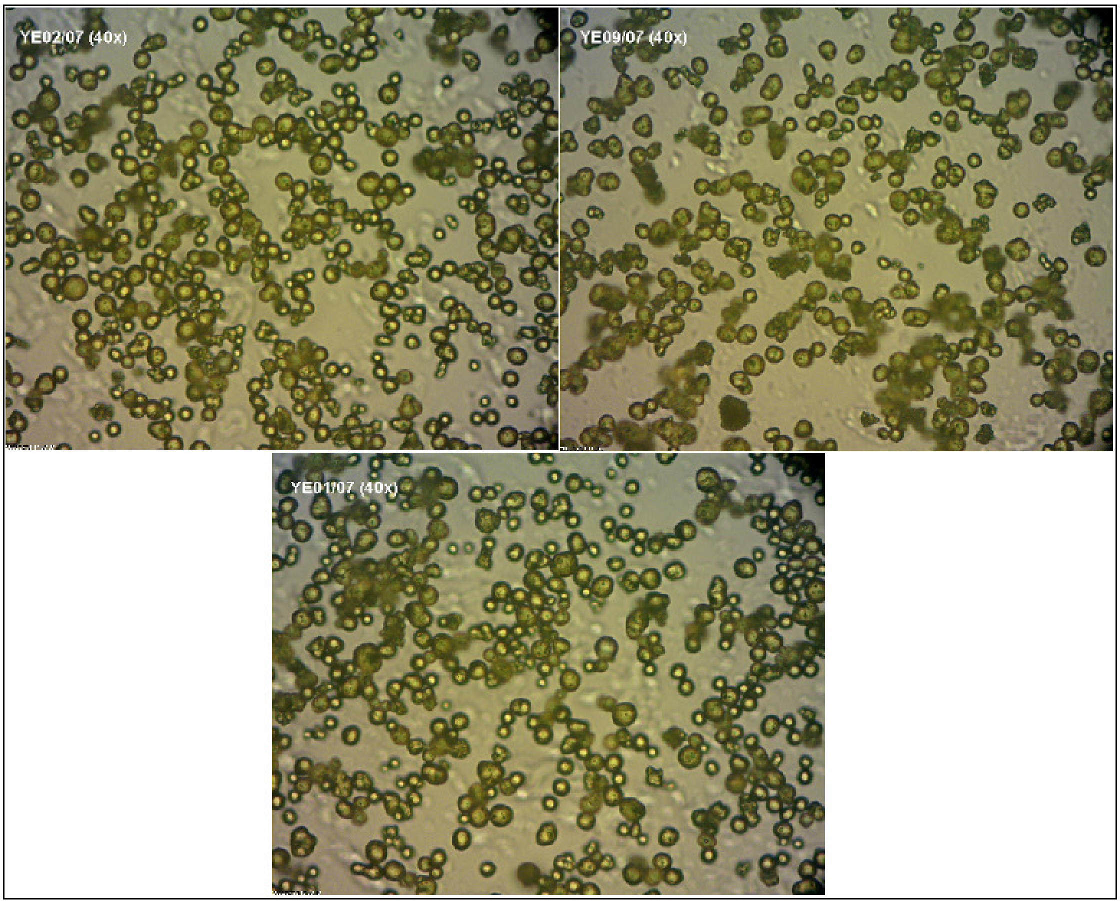

Figure 3.7: The microscopic pictures for toner particles series VII

From the results shown in Table 3.20, all the properties were quite consistent as the percentage of wax will not affect the other parameters. There is a very obvious trend in the wax content as the percentage of wax used varied shown in the DSC spectrum. From the result, it can be concluded that about 1% of the total wax put in will not be incorporated into the toner particles and was washed out during washing and filtering process. Figure 3.7 showed that the fusing efficiency improve a lot with more amount of waxes incorporated in as the wax serves as a plasticizer that can reduce the T_g of the resin.

Table 3.20: The properties summary of raw toners series VII

Properties		**Average Value Measured**			**Units**
		YE02/07	**YE09/07**	**YE01/07**	
Particle Size Distribution (PSD)	**D5**	0.78	0.81	0.79	**µm**
	D50	6.02	6.65	6.28	**µm**
	D95	17.67	15.44	15.11	**µm**
	5 µm	32.62	26.41	29.19	**%**
	24 µm	98.14	99.71	99.69	**%**
Sharpness Index		22.65	22.65	19.06	**-**
Tribocharge		-7.3	-7.0	-7.5	**µC/g**
Relative Dielectric Constant.		2.20	2.17	2.18	**-**
Dissipation Factor		125.0	119.6	125.4	**-**
Melt Flow Index at 130°C		70.23	72.31	73.21	**g/10 min**
Glass Transition Temperature, T_g		55	53	52	**°C**
Wax Melting Point, T_p		76	76	76	**°C**
Wax Content		**4.88**	**5.82**	**6.89**	**%**
Molecular Weight Distribution (MWD)	**M_n (10^4)**	0.94	1.30	0.94	**g/mol**
	M_w (10^4)	2.62	2.89	2.83	**g/mol**
	M_p (10^4)	2.05	2.09	2.11	**g/mol**
	M_w/M_n	2.79	2.22	3.00	**-**

3.4.8 Effect of latexes prepared using different percentage of CTA

All the other parameters were maintained as shown in the Table 3.21 below except the latexes used were varied. The latexes used were prepared with different percentage of chain transfer agent as mentioned in Chapter 2, section 2.2.

Table 3.21: Raw materials composition in weight (g) used for series VIII

Sample ID	**Latex of different % of CTA**		**Pigment Dispersion (g) (TSC = 20%)**	**Wax Dispersion (g) (TSC = 35%)**	**Distilled Water (g)**	**PAC (g) (10% w/w solution)**
	Latex Code	**Weight (g)**				
YE01/07	**E125/09**	**138.01**	15.00	13.70	280.35	2.00
ME28/07	**E127/09**	**137.98**	15.00	13.70	280.35	2.00
YE10/07	**E128/09**	**138.01**	15.00	13.70	280.35	2.00
YE11/07	**E129/09**	**138.38**	15.00	13.70	280.35	2.00
YE12/07	**E130/09**	**138.23**	15.00	13.70	280.35	2.00
YE43/07	**E149/09**	**137.88**	15.00	13.70	280.35	2.00

With the reference to Table 3.22, the melt flow index reduced as the latex of lower percentage of chain transfer agent was used in a predicted manner. The T_g and the molecular weight distribution increased as the chain transfer agent in the latex reduced. The chain transfer agent worked as a "modifier" especially in emulsion polymerization to lower the average molecular weight distribution of the copolymer as well as the T_g of the coplymer. As for the T_g of these raw toners also slightly reduced relatively to the T_g of the copolymer latex itself because the wax served as a "plasticizer" as mentioned earlier.

The melt flow index of the raw toner also increases slightly because of the presence of the wax. The other properties do not show obvious significant.

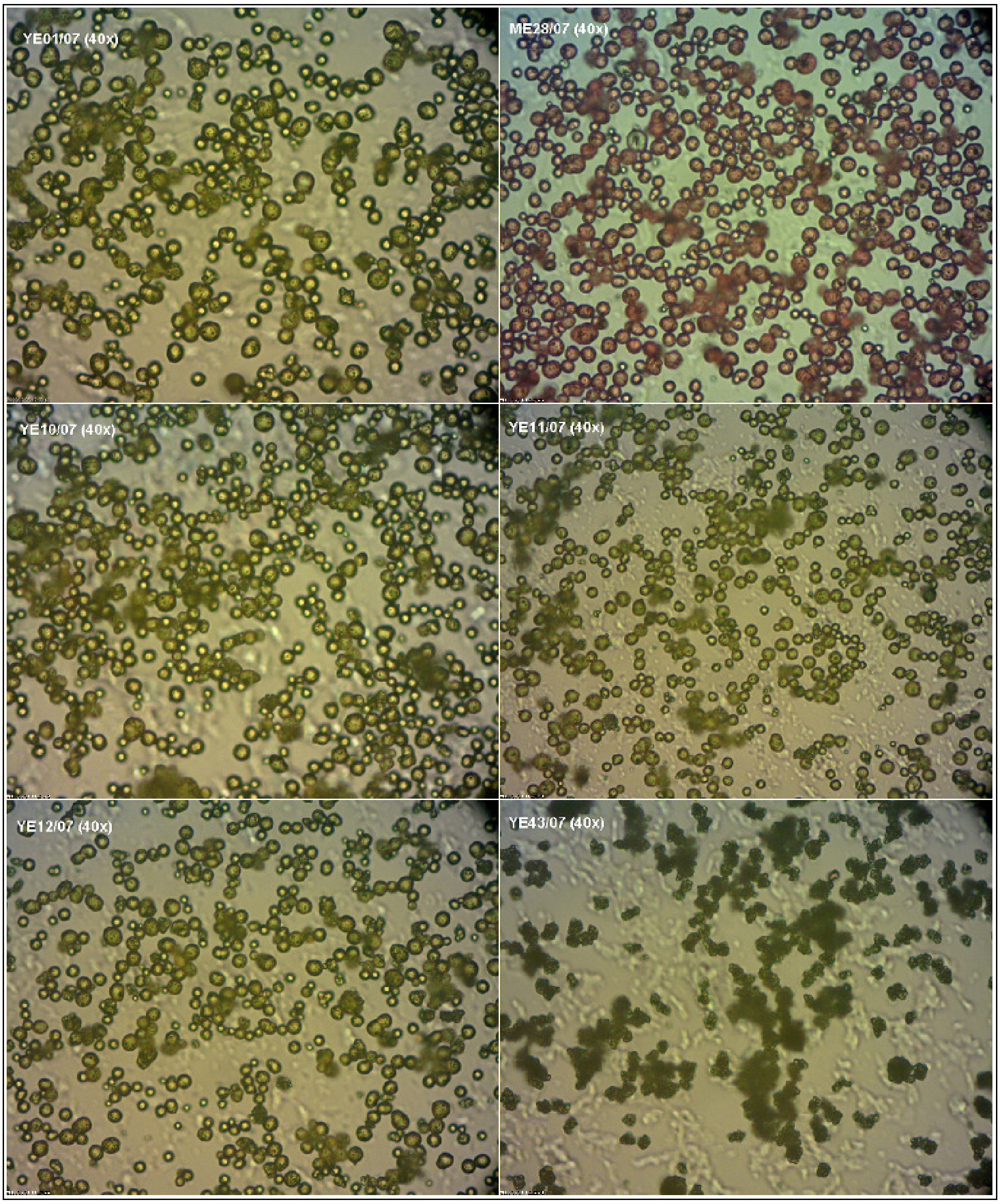

Figure 3.8: The microscopic pictures for toner particles series VIII

Table 3.22: The properties summary of raw toners series VIII

Properties		**Average Value Measured**						**Unit**
		YE01/07	**ME28/07**	**YE10/07**	**YE11/07**	**YE12/07**	**YE43/07**	
Particle Size Distribution (PSD)	**D5**	0.79	0.79	0.72	0.64	0.72	0.92	**µm**
	D50	6.28	5.82	5.21	4.62	5.21	4.93	**µm**
	D95	15.11	16.64	13.68	8.24	18.10	11.67	**µm**
	5 µm	29.19	35.26	45.57	58.82	46.35	51.11	%
	24 µm	99.69	99.60	99.79	100.00	98.58	98.58	%
Sharpness Index		19.13	21.06	19.0	12.88	25.14	19.21	**-**
Tribocharge		-7.0	-8.0	-6.5	-7.8	-7.5	-7.5	**µC/g**
Relative Dielectric Constant.		2.20	2.16	2.30	2.20	2.29	2.41	**-**
Dissipation Factor		120.5	122.1	130.0	128.7	121.0	130.8	**-**
Melt Flow Index at 130°C		**75.22**	**65.50**	**60.12**	**40.65**	**30.21**	**18.46**	**g/10 min**
Glass Transition Temperature, T_g		**52.49**	**53.29**	**53.89**	**55.26**	**55.48**	**58.83**	°C
Wax Melting Point, T_p		75.55	75.36	75.48	75.86	75.53	75.41	°C
Wax Content		6.89	6.53	6.79	6.30	6.10	5.29	%
Molecular Weight Distribution	**M_n (10^4)**	**0.94**	**1.08**	**1.37**	**1.32**	**1.41**	**1.65**	**g/mol**
	M_w (10^4)	2.83	3.64	2.65	2.77	2.65	3.61	**g/mol**
	M_p (10^4)	2.11	2.48	2.19	2.13	4.43	3.04	**g/mol**
	M_w/M_n	3.00	3.36	1.93	2.11	1.89	2.20	**-**

3.5 Latex Blending

A series of latexes blending was carried out with latex E130/07 and E149/08 in different ratio. The reason these two latexes were chosen to do blending out of all the other latexes is because the copolymers isolated out from these two latexes have the MFI of 24.84 g/10 min and 16.58 g/10 min respectively which believed will give the average MFI value of the copolymer isolated from the latex blending in range of 20.00 g/10 min to 25.00 g/10 min which is the range of MFI found in the commercial toner available in the market. The properties of all the latexes blended are shown in Table 3.23.

Table 3.23: The summary of all the properties measured on blended latexes

Properties of the copolymers		**Ratio E130/07 latex to E149/08 latex**							**Unit**
		20:80	**30:70**	**40:60**	**50:50**	**60:40**	**70:30**	**80:20**	
Total Solid Content		37.91	38.02	38.12	38.25	37.89	38.24	38.10	%
Melt Flow Index at 130°C		**22.21**	**23.14**	**23.02**	**25.01**	**29.02**	**32.24**	**38.07**	**g/10min**
Glass Transition Temperature, T_g		**63**	**62**	**62**	**60**	**60**	**60**	**58**	°C
Molecular Weight Distribution	**M_n (10^4)**	1.94	1.46	1.49	1.70	1.78	1.63	1.51	**g/mol**
	M_w (10^4)	3.93	3.59	3.29	3.89	3.86	4.51	3.65	**g/mol**
	M_p (10^4)	3.40	3.16	2.96	2.84	2.93	2.86	2.64	**g/mol**
	M_w/M_n	2.03	2.45	2.21	2.29	2.18	2.77	2.42	**-**

The melt flow index shows an increasing trend as the ratio of E130/07 latex to E149/08 latex increased. On the other hand, the glass transition temperature shows a decreasing trend as predicted.

3.6 Preparation of raw toner using EA process using blended latex

In the emulsion-aggregation process, the latexes of E130/07 and E149/08 in ratio 50:50 were used throughout the experiments because the latex blending properties showed the desired MFI and T_g. For these 3 batches of blending, the results were measured in the finished toner stage instead of the raw toner stage because results from all the previous experiments, the properties like particle size distribution (PSD), sharpness index, relative dielectric constant, dissipation factor, melt flow index (MFI), glass transition temperature (T_g), wax melting point (Tm), wax content and molecular weight distribution do not differ much between raw toner and finish toner. The only one properties that have obvious differences will be the tribocharge as the finished toner will always have higher tribocharge than the raw toner by a -10 to -5 μC/g because of the addition of silica that can improve the smoothness of the particle surface and resulted in more uniform charge. Thus, the final properties of the finished toner will be shown in the next part instead of the raw toner.

3.7 Additive Blending

After the additive blending, the finished toners were used to performance test print and performance test. The properties results of the finished toners are shown in Table 3.24.

Table 3.24: The summary of all the properties measured on finished toner

Properties		**Average Value Measured**		**Unit**
		KFT01/08	**KFT06/08**	
Particle Size Distribution (PSD)	**D5**	1.06	0.93	**µm**
	D50	6.04	5.56	**µm**
	D95	13.04	9.47	**µm**
	5 µm	36.29	38.56	**%**
	24 µm	99.82	100.00	**%**
Sharpness Index		12.30	10.18	**-**
Tribocharge		**-17.5**	**-20.0**	**µC/g**
Melt Flow Index at 130°C		23.78	24.78	**g/10 min**
Flowability		5	5	**mm**
Apparent Density		0.33	0.32	**g/m^3**
Glass Transition Temperature, T_g		55.59	54.62	**°C**
Wax Melting Point, T_p		76.43	76.07	**°C**
Wax Content		5.82	6.30	**%**
Molecular Weight Distribution	**M_n (10^4)**	1.89	1.81	**g/mol**
	M_w (10^4)	5.40	4.43	**g/mol**
	M_p (10^4)	2.92	2.85	**g/mol**
	M_w/M_n	2.85	2.45	**-**

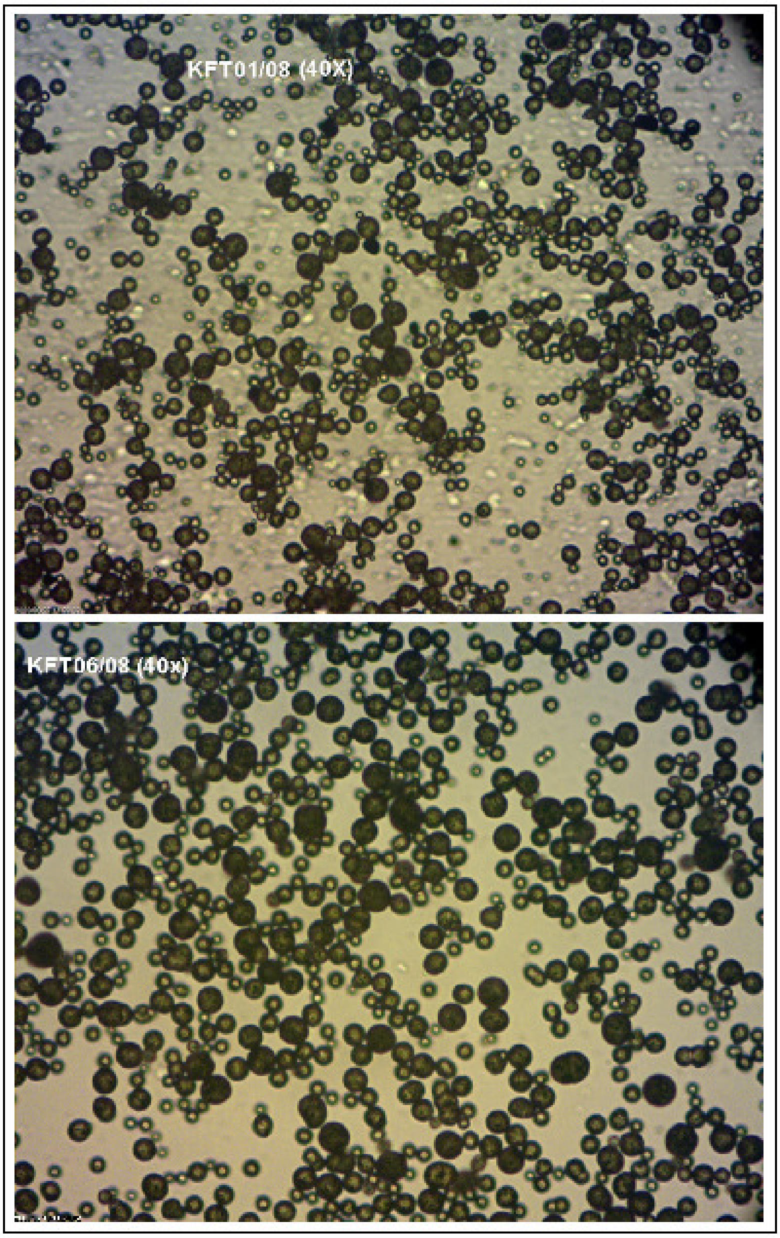

Figure 3.9: The microscopic pictures for cases KFT01/08 and KFT06/08

Overall, the properties shown in Table 3.25 of both finished toner are quite consistent except for the tribocharge. Case KFT06/08 has higher tribocharge that KFT01/08 because the blending time for KFT06/08 was longer than KFT01/08. Besides contributing to flowabilty of the toner, silica is also known to contribute uniform tribocharge to the surface of the finished toner. Hence, sufficient time is required for additive blending so that the silica can coat evenly on the surface of the raw toner to produce finished toner with more even charge. The toner particles fused very well for both cases where more than 95% particles were fused with spherical shape as shown in Figure 3.9.

The test print on KFT01/08 was very bad where the colour tone was very light and uneven with a lot of white spots as shown in test chart (Appendix E). This could due to the relatively low tribocharge that caused the finished toners were unable to coat on the photoreceptor drum properly during printing. The performance test did not continue any further for this case.

The test print on KFT06/08 improved a lot as compared to case KFT01/08. The color tone is much darker with even distribution. The starting printout did show a bit of different tone but as the printing goes on, the color tone became very even. The performance test continued to the end of the cycle of 2100 copies. The test chart of the printout was very consistent until 2100 copies. The full set of test chart and performance test are shown in Appendix F to I.

3.8 Comparison of the commercial after market CPT

There are various after market chemically produced toners in the markets beside the Original Equipment Material (OEM). The similar characterization was done on these toners to use as the standard to compare with the toners that was prepared. Table 3.25 and

Table 3.26 below are the summary of all the properties for after market raw toners from three different manufacturers, Avecia from Fuji Film, Static Control Color (SCC) and HP Original Equipment Manufacturer (OEM) for model 2600 and 1515. The microscope pictures were also taken to show the fusing of these OEM and after market toners in Figure 3.10 to Figure 3.12. Table 3.27 is the comparison of the black colour finished toner compared to the black finished toner from only the OEM since the other after market toner manufacturer such as SCC and Avercia do not produce black toner.

From the properties checked, the after market toners showed a very wide range in all the properties. The particle size distribution varies from 6.50 to 7.50 μm. The M_w is in the range of 2.00 to 9.00 x 10^4 g/mol, M_n in the range of 1.00 to 3.50 x 10^4 g/mol, M_p in the range of 1.90 to 5.60 x 10^4 g/mol and the polydispersity in the range of 1.60 to 3.10. The melt flow index is in the range of 10.0 to 30.0 g/10 min. As for the T_g, is in the range of 52-57°C. The melting point for the waxes are in quite a big range from 70 to almost 100°C as these manufacturer might be using different waxes as there are so many types of waxes available in the market. Thus, the wax content also differs quite a lot in the range of 1.80 to 8.70%.

As for the fusing, the OEM toners are more of the spherical shape and fused almost 100% whereas for the Avercia and SCC toner, mostly are in ‘potato’ shapes with mixture of spherical shapes.

Table 3.25: The properties summary of commercial toners from original equipment manufacturer (OEM)

Properties		**HP Color LJ 2600n (OEM)**				**HP CP1515 Color (OEM)**				**Units**
		Cyan	**Yellow**	**Magneta**	**Black**	**Cyan**	**Yellow**	**Magenta**	**Black**	
Particle Size Distribution (PSD)	**5%**	0.80	0.82	0.85	4.93	0.82	0.75	0.87	3.77	**µm**
	50%	7.16	6.70	6.94	8.09	5.31	5.43	5.93	5.58	**µm**
	95%	11.67	10.95	11.45	13.79	8.46	9.26	10.80	8.70	**µm**
	5 µm	16.69	20.79	19.65	5.31	41.88	40.74	33.41	34.09	**%**
	24 µm	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	**%**
Sharpness Index, D95/D5		14.59	13.35	13.47	2.80	10.32	12.35	12.41	2.31	-
Molecular Weight Distribution	**Mw ((10^4)**	7.36	7.99	8.90	7.89	3.18	3.23	3.18	3.58	**g/mol**
	Mn (10^4)	2.93	2.99	2.92	2.94	7.36	6.64	6.25	8.20	**g/mol**
	Mp (10^4)	3.65	37791	36529	3.74	3.84	3.99	3.92	3.89	**g/mol**
	Polydispersity	2.51	2.68	3.05	2.69	2.31	2.05	1.97	2.29	**-**
Tribocharge		-16.00	-23.25	-19.25	-22.00	-27.0	-26.0	-25.0	-26.5	**µC/g**
Relative Dielectric Constant		2.23	2.18	2.20	2.52	The machine was out of service				**-**
Dissipation Factor		130.8	236.7	120.6	150.0					**-**
Melt Flow Index at 130°C		18.25	19.21	17.79	21.54	17.65	18.21	17.04	37.60	**g/10 min**
Flowability (Disc method)		4	4	4	5	5	5	5	5	**mm**
Apparent Density		0.491	0.485	0.473	0.504	0.396	0.415	0.420	0.402	**g/cm**
Glass Transition Temperature (T_g)		53	52	53	57	56	56	56	56	**°C**
Wax Melting Point (Tp)		72.31	72.48	72.48	72.02	77.72	77.58	77.74	77.34	**°C**
Wax Content		6.18	6.17	6.62	4.39	5.35	5.51	5.82	7.00	**%**

Table 3.26: The properties summary of commercial after market toners from other manufacturers

Properties		**HP Color LJ 2600 (SCC)**			**HP Color LJ 2600 (Avercia)**			**Unit**
		Cyan	**Yellow**	**Magenta**	**Cyan**	**Yellow**	**Magenta**	
Particle Size Distribution (PSD)	**5%**	0.75	0.82	0.79	0.91	0.79	0.87	**µm**
	50%	7.06	6.73	6.63	7.50	6.71	7.34	**µm**
	95%	11.59	12.24	10.95	11.96	10.94	13.15	**µm**
	5 µm	17.88	22.89	22.57	12.75	20.19	18.06	**%**
	24 µm	100.00	100.00	100.00	100.00	100.00	100.00	**%**
Sharpness Index, D95/D5		15.45	14.93	13.86	13.14	13.85	15.11	**-**
Molecular Weight Distribution	**Mw (10^4)**	6.35	5.84	6.81	2.35	2.25	2.30	**g/mol**
	Mn (10^4)	3.44	3.30	3.23	1.35	1.35	1.33	**g/mol**
	Mp (10^4)	5.61	5.19	5.17	1.99	1.92	1.95	**g/mol**
	Polydispersity	1.85	1.77	2.11	1.74	1.66	1.72	**-**
Tribocharge		-24.50	-29.00	-24.00	-26.25	-26.75	-24.75	**µC/g**
Relative Dielectric Constant		2.36	2.54	2.75	2.44	2.38	2.34	**-**
Dissipation Factor		138.4	145.8	84.2	86.7	98.0	92.1	**-**
Flowability (Disc method)		4	4	4	4	4	4	**mm**
Melt Flow Index at 130°C		25.24	23.54	26.28	*13.5 (160°C)	*13.3 (160°C)	*14.8 (160°C)	**g/10 min**
Glass Transition Temperature (T_g)		58	55	55	55	57	52	**°C**
Wax Melting Point (Tp)		98.81	98.62	98.47	80.56	85.37	78.90	**°C**
Wax Content		8.30	8.56	8.61	2.63	2.79	1.89	**%**

* Measurement of MFI was measured at 160°C instead of 130°C because the MFI for these samples are too low to be measured with 130°C

Table 3.27: The properties comparison of the prepared black finished toner with OEM

Properties		**OEM**	**KFT05/08**	**KFT06/08**	**Unit**
Particle Size Distribution (PSD)	**5%**	3.77	1.06	0.93	**µm**
	50%	5.58	6.04	5.56	**µm**
	95%	8.70	13.04	9.47	**µm**
	5 µm	34.09	36.29	38.56	%
	24 µm	100.00	99.82	100.00	%
Sharpness Index, D95/D5		2.31	12.30	10.18	-
Molecular Weight Distribution	**Mw (10^4)**	3.58	1.89	1.81	**g/mol**
	Mn (10^4)	8.20	5.40	4.43	**g/mol**
	Mp (10^4)	3.89	2.92	2.85	**g/mol**
	Polydispersity	2.29	2.85	2.45	**-**
Tribocharge		-26.5	-17.5	-20.0	**µC/g**
Flowability (Disc method)		5	5	5	**mm**
Melt Flow Index at 130°C		37.60	23.78	24.78	**g/10 min**
Glass Transition Temperature (T_g)		56	56	55	**°C**
Wax Melting Point (Tp)		77.34	76.43	76.07	**°C**
Wax Content		7.00	5.82	6.30	%

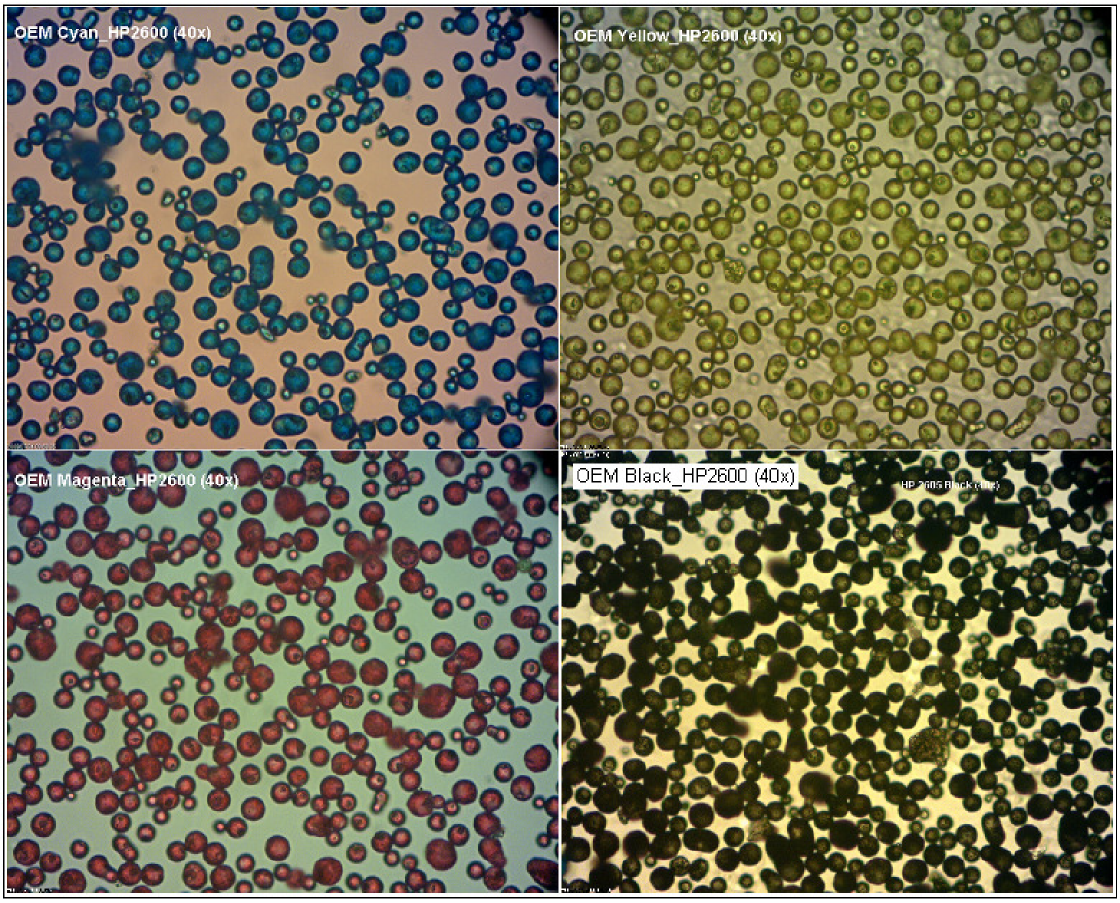

Figure 3.10: The microscope pictures of OEM finished toners HP2600 series

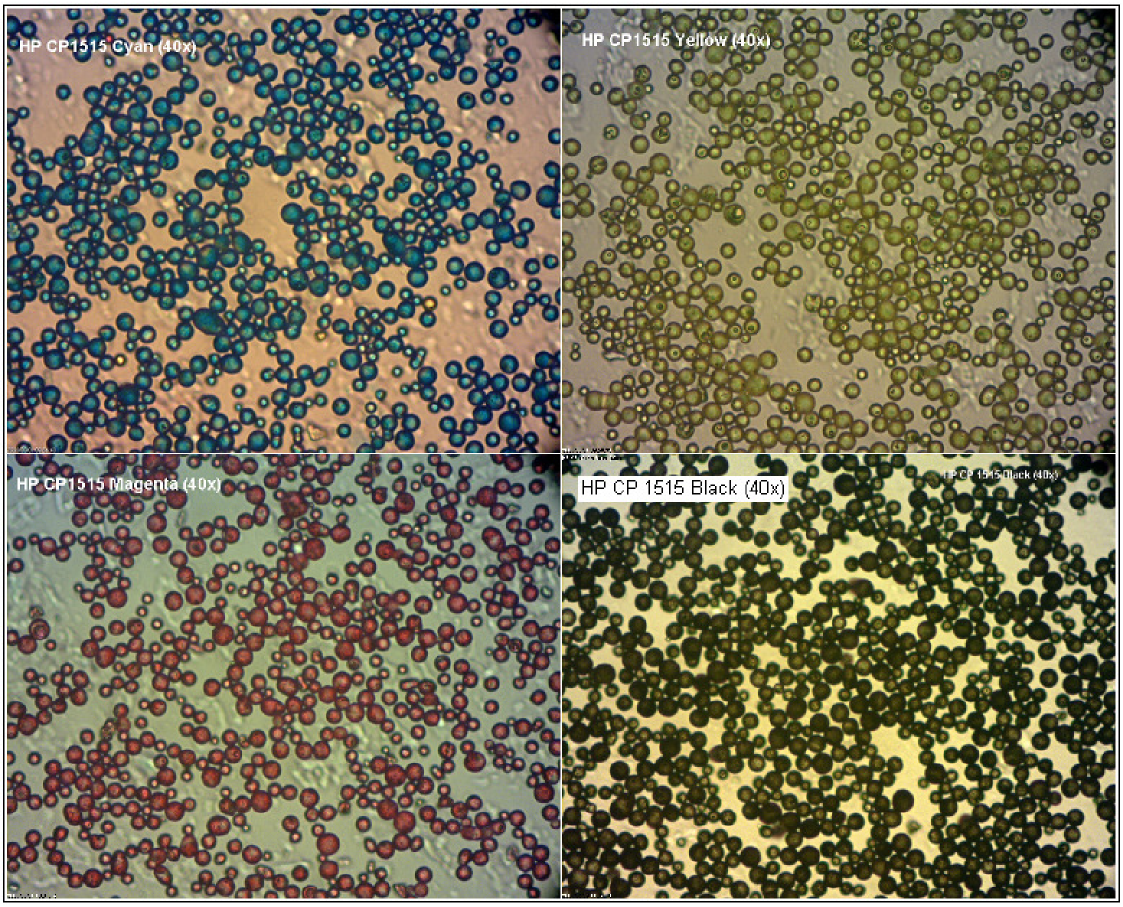

Figure 3.11: The microscope pictures of OEM finished toners HP1515 series

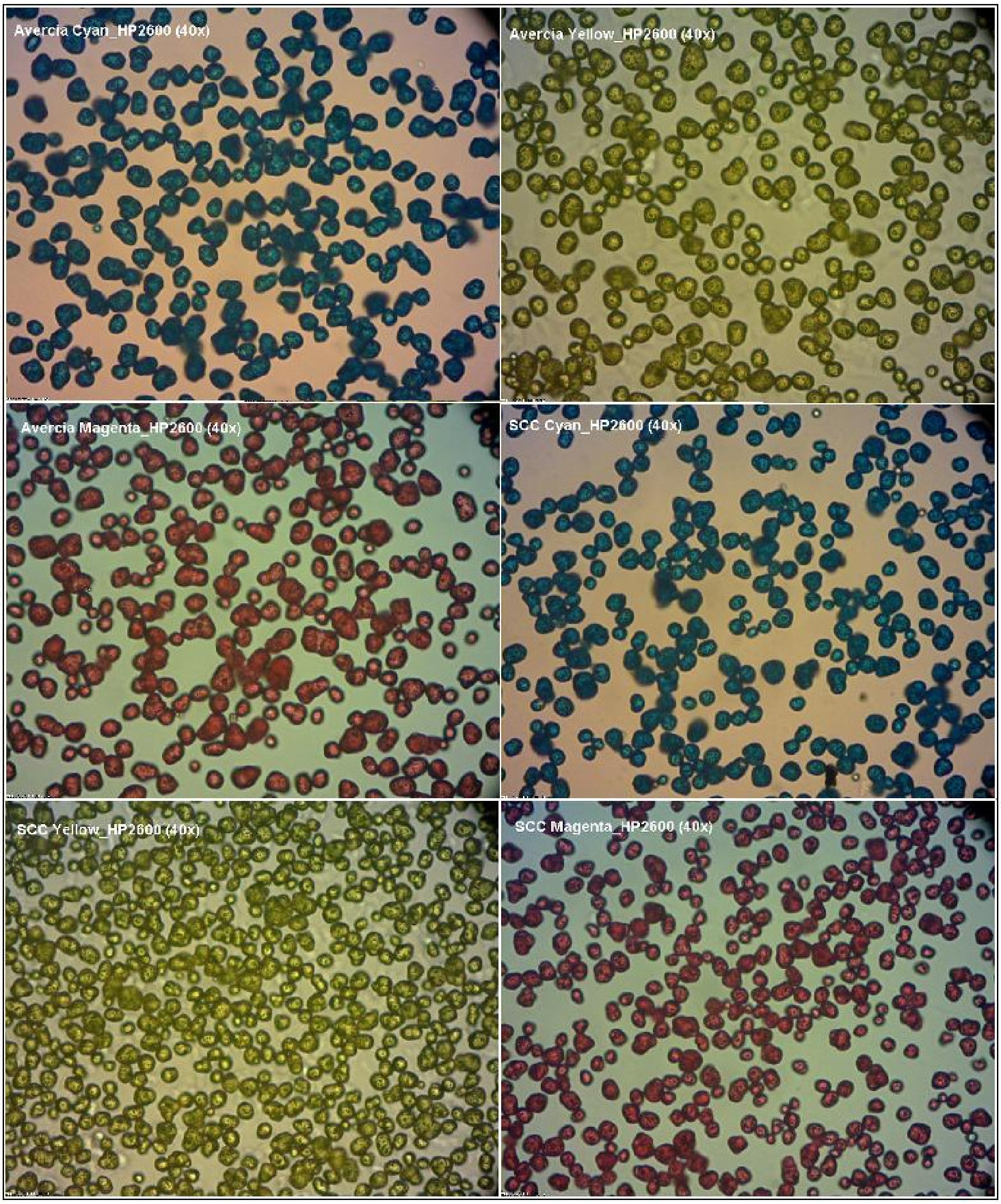

Figure 3.12: The microscope pictures of other manufacturer (Avercia and SCC) CPT tone