Chapter 3

Methodology and Experimentation

3.1 Experimental Details

This chapter deals with the study mine description, haul dust sample collection, screening, sieving, chemicals, polymerization process, equipment and other instruments used for the laboratory study. Also generalized information regarding the experimentation of moisture content of dust and its emission rate are discussed.

3.2 Mine Description

Dust sample was collected from a haul road of an Opencast Coal Mine, under the Block-II area of Bharat Coking Coal Limited (BCCL) in Dhanbad district of Jharkhand state in India. The mine is situated about 40 km west of Dhanbad town. Block-II area came into existence in the year 1983-84. Coking coal is the prime product of this area. A schematic diagram is presented in Figure 3.1 for better visualization of the mine area in Indian map.

3.2.1 Climatic condition

The climate of the study mine area is sub-humid, sub-tropical monsoonal type. Summer starts from the middle of March and continues till nearly end of June. The atmospheric temperature, humidity and pressure variation of June, the hottest period, and January, the coldest period,

of last four years 2017, 2018, 2019 & 2020 are given in the Table 3.1. The average annual precipitation is 1200-1400 mm.

Table 3.1 Weather reports of Dhanbad district from 2017 to 2020

Date	Temperature (°C)			Humidity (%)			Pressure (mbar)		
	Lowest	Highest	Average	Lowest	Highest	Average	Lowest	Highest	Average
June 2020	23 °C	43 °C	33 °C	22%	99%	69%	991	1008	999
January 2020	4 °C	29 °C	18 °C	09%	97%	61%	1007	1020	1014
June 2019	24 °C	48 °C	32 °C	11%	95%	66%	986	1005	999
January 2019	2 °C	30 °C	18 °C	14%	100%	63%	1012	1022	1017
June 2018	25 °C	42 °C	31 °C	23%	94%	68%	990	1006	998
January 2018	8 °C	30 °C	17 °C	14%	93%	59%	1006	1019	1013
June 2017	22 °C	42 °C	31 °C	37%	100%	71%	993	1009	999
January 2017	8 °C	31 °C	19 °C	10%	96%	62%	1008	1021	1015

Figure 3.1 (a): Map of India, (b): Map of Dhanbad district in Jharkhand state, (c): Map location of Block II study area

3.3 Methods of Haul Road Dust Sample Collection

Upper layer of haul road dust particles are generally liable to become airborne. Due to regular running of heavy vehicles, road surface materials are continually crushed and pulverised in to small pieces. Upper surface of 10–15 mm depth is made of silt and tiny particles. Below this depth, parent soil or gravel material is in the original lump size. The percentage of coal in haul roads near the coal face is, in general, very high, whereas it is minimal on the haul

roads of overburden dump sites. So it was tried to get a sample of dust which is a good representation for the whole haul road. It was preferred to take a section of 100 m length at the middle of haul road of mine. A total of 20 points at equal distance were marked in 100 m length. Hand-pick big boulders and gravels were discarded. A sample of 100 g was taken from each of the marked points. Thus, total 2 kg of dust sample was collected from this section of the haul road and put into a clean plastic bag. The bag was sealed and brought to the laboratory for the research work. Sample was put into an oven at 110 °C for 120 h duration for drying it completely.

3.4 Chemical Materials

Acrylamide (AM), amylopectin (AP), guar-gum (GG), 2-acrlyamido-2-methylpropanesulfonic acid (AMPS), ceric ammonium nitrate (CAN), nitrogen (N_2) gas, hydroquinone and acetone were procured from S.D. Fine Chemicals, Mumbai, India. All the chemicals were used in the study without any further purification. Distilled water was used for conducting the experiments.

3.5 Synthesis of Polymers

All the polymers were synthesized in the laboratory by free radical polymerization reaction mechanism which has been discussed in section 2.13. All the synthesized polymers were packed in airtight glass vials and preserved in a desiccator.

3.5.1 Synthesis of Polyacrylamide (PAM)

The acrylamide monomer of 5 g was dissolved in 20 ml of distilled water in a conical flask of 100 ml (Figure 3.2). The solution was stirred for 30 min in nitrogen atmosphere (N_2 gas). 20 mg of ceric ammonium nitrate (CAN) was mixed with the solution as initiator, N_2 purging was done for the next 30 min at 50 °C. A Schematic representation of chemical reactions taking place in PAM synthesis is shown in Figures 3.3 & 3.4. Magnetic stirring of the mixture was continued till the solution became viscous. It was then left for cooling at room temperature. The reaction was terminated by adding 2 ml of a saturated solution of hydroquinone. The product was precipitated by using acetone as a non-solvent. The resultant polymer lumps were broken into small pieces by hand. It was dried in the oven for 24 h at 70 °C and then pulverized in a grinder. The granular PAM was packed in glass vials (Figure 3.5) and preserved in a desiccator.

Figure 3.2 Chemical reaction set-up in conical flask with N_2 purging and stirring

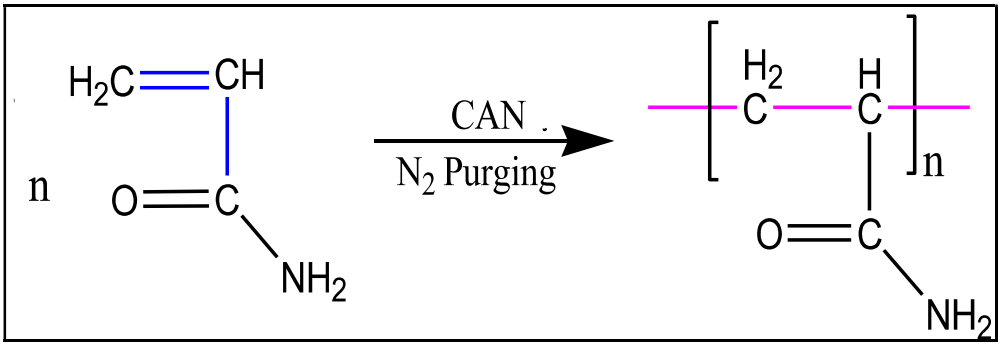


Figure 3.3 Polymerization reaction process of polyacrylamide

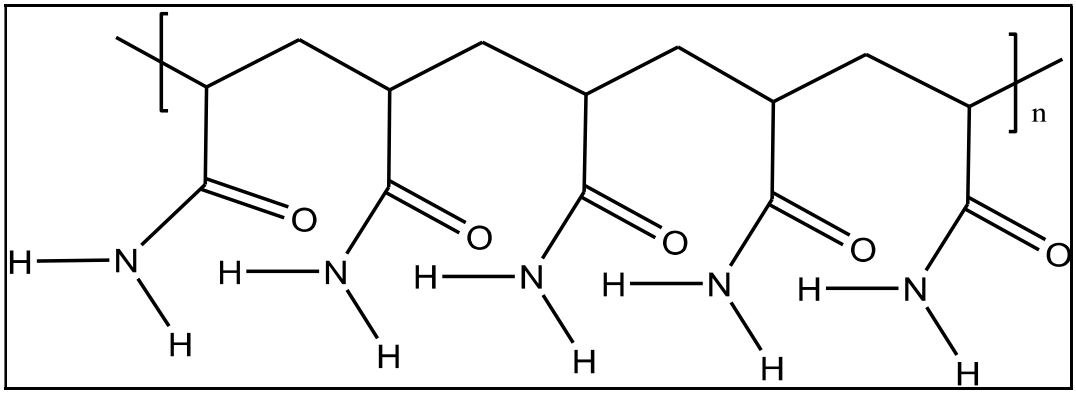


Figure 3.4 Chemical structure of polyacrylamide

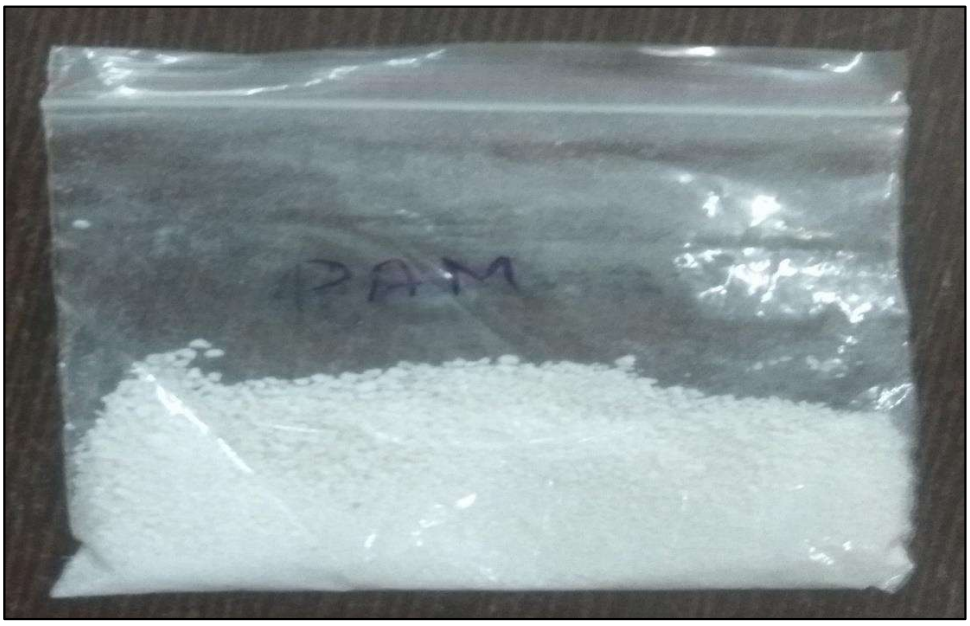


Figure 3.5 Granular PAM product

3.5.2 Synthesis of Hydrolyzed Polyacrylamide (H-PAM)

The synthesis of H-PAM was done by hydrolysis of PAM with NaOH solution (Figure 3.6). The entire process of PAM synthesis is given in the previous section. 1 g of previously synthesized PAM was dissolved in a 400 ml of distilled water in a beaker of 1000 ml by electronic magnetic stirring at 50 °C. The reaction was continued until PAM dissolved properly. Thereafter, 20 ml of 1N NaOH solution was mixed. Then the reaction mixture was stirred for 3 hours. The product was precipitated by using acetone as a non-solvent. Rest of the procedure is same as the synthesis of PAM. Finally, it was packed and preserved in a desiccator. It may be mentioned that only partial hydrolysis takes place in this process. The resultant polymer contains both amide and carboxylic functionalities.

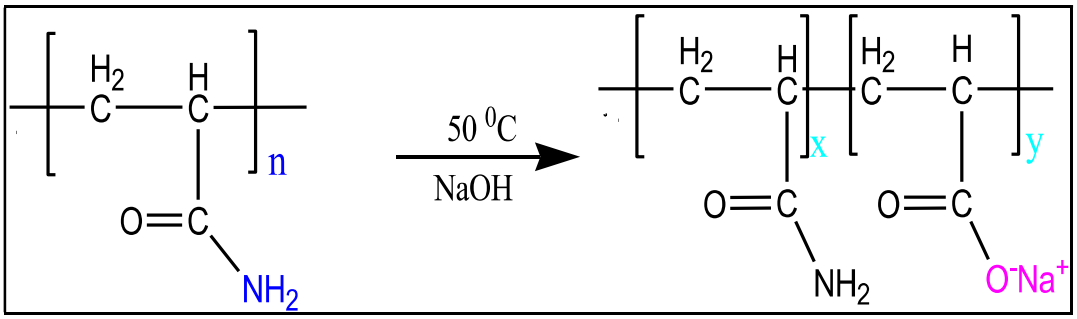
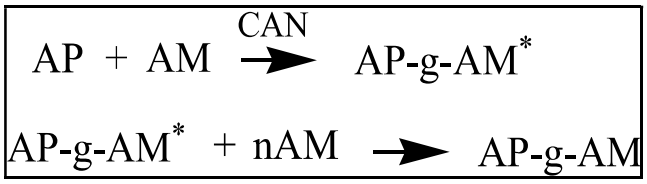


Figure 3.6 Polymerization reaction process of H-PAM

3.5.3 Synthesis of Amylopectin-grafted-Polyacrylamide (AP-g-PAM)

Amylopectin based graft polyacrylamide polymer was synthesized by free radical polymerization technique using CAN initiator (Figure 3.7) [89]. Synthesis process of AP-g-PAM is presented in Figure 3.8. 2 g of acrylamide was dissolved in 20 ml distilled water in

a conical flask. Then 1 g of AP was dissolved in 400 ml of distilled water and mixed with acrylamide solution. The flask was placed in a water bath at 60 °C and nitrogen (N_2) was purged for 30 min. Thereafter, 20 mg of CAN solution was added to the reactants. N_2 gas was purged yet again for 30 min. Then supply of N_2 gas was stopped, and the reaction was allowed to continue for 24 h. After that, the reaction was terminated by adding 2 ml of a saturated solution of hydroquinone. The final product was precipitated by acetone. The lump product was broken into small pieces and made powder by grinding. It was dried in a oven at 70 °C for 24 h. Finally it was stored in the glass vials.



* represents radical and n represents multiple repetitions

Figure 3.7 Reaction steps of AP-g-PAM polymerization

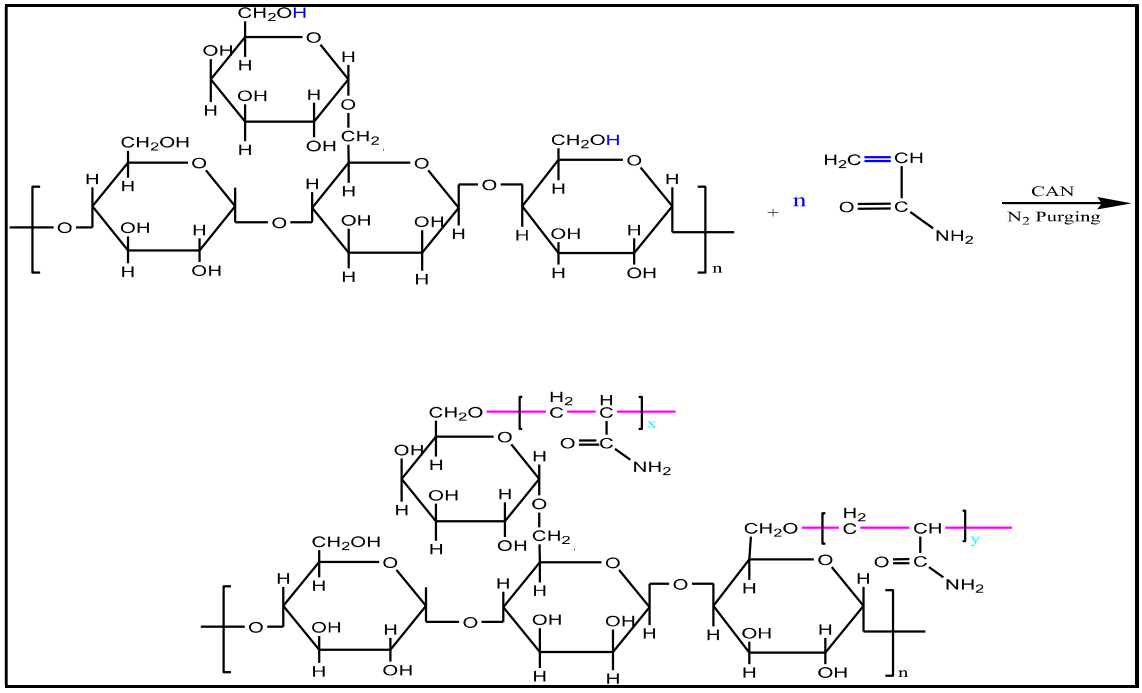


Figure 3.8 Polymerization reaction process of AP-g-PAM

3.5.4. Synthesis of Hydrolyzed-Amylopectin-grafted-Polyacrylamide (H-AP-g-PAM)

The synthesis of H-AP-g-PAM was done through hydrolysis of AP-g-PAM with NaOH solution (Figure 3.9). The process of AP-g-PAM synthesis was already elaborated in the previous section. 1 g of AP-g-PAM was dissolved in a 500 ml distilled water in a beaker of 1000 ml by electronic magnetic stirring at 50 °C. The reaction was continued till the polymer dissolved properly. After that 20 ml of 1N NaOH solution was added. Then the reaction mixture was stirred on the magnetic stirrer up to 3 hours. Rest of the process to obtain H-AP-g-PAM is similar to the process of H-PAM.

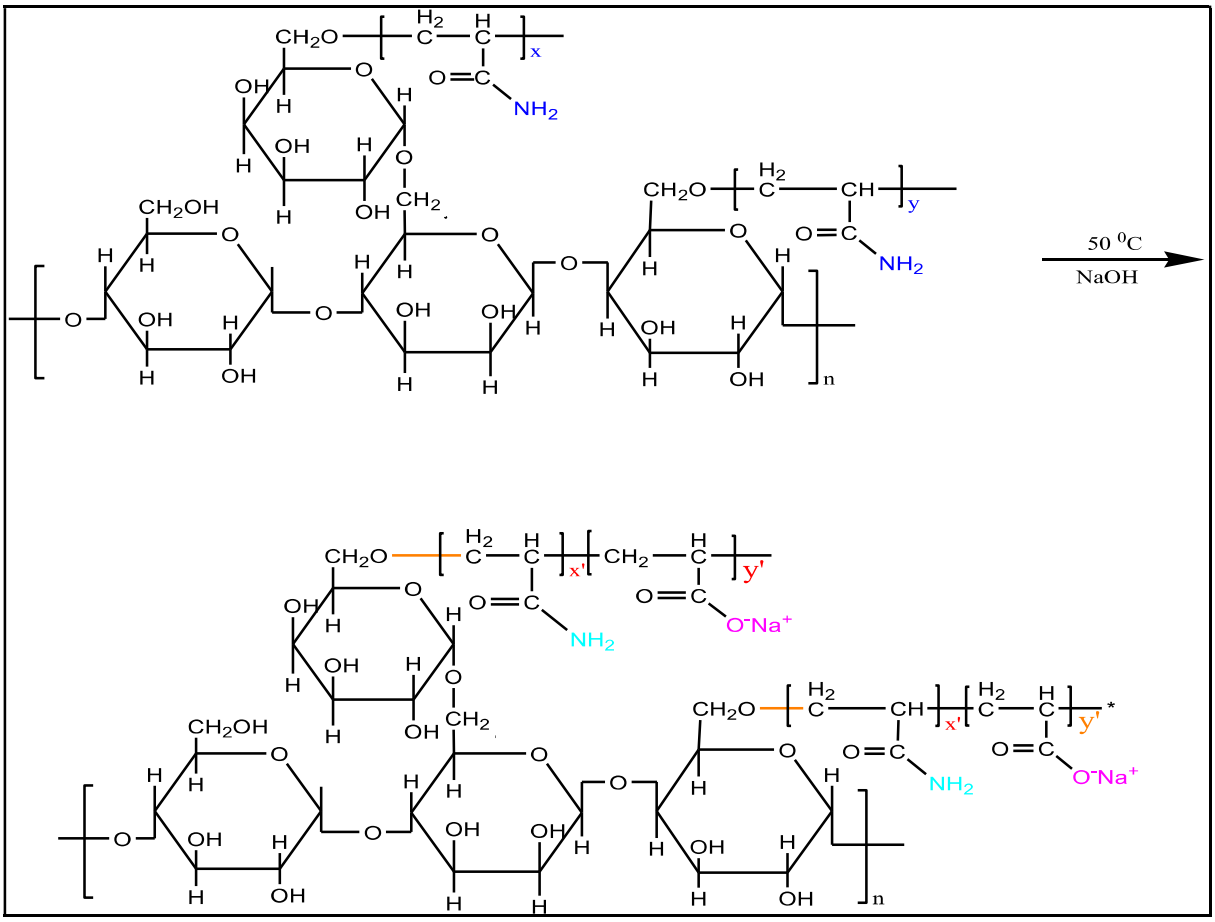


Figure 3.9 Polymerization process of H-AP-g-PAM

3.5.5. Synthesis of Guargum-grafted- Polyacrylamide (GG-g-PAM)

Guargum based graft polyacrylamide polymer was synthesized by free radical polymerization technique using CAN initiator (Figure 3.10) [90]. 2 g of acrylamide was dissolved in 20 ml distilled water in a conical flask. Then 1 g of GG was dissolved in 400 ml distilled water and mixed with acrylamide solution. The flask was placed in a water bath at 60 °C and nitrogen (N_2) is purged for 30 min. Thereafter, 20 mg of CAN solution was added to the reactants. N_2 gas was purged yet again for 30 min. Rest of the procedure is similar to the synthesis of AP-g-PAM.

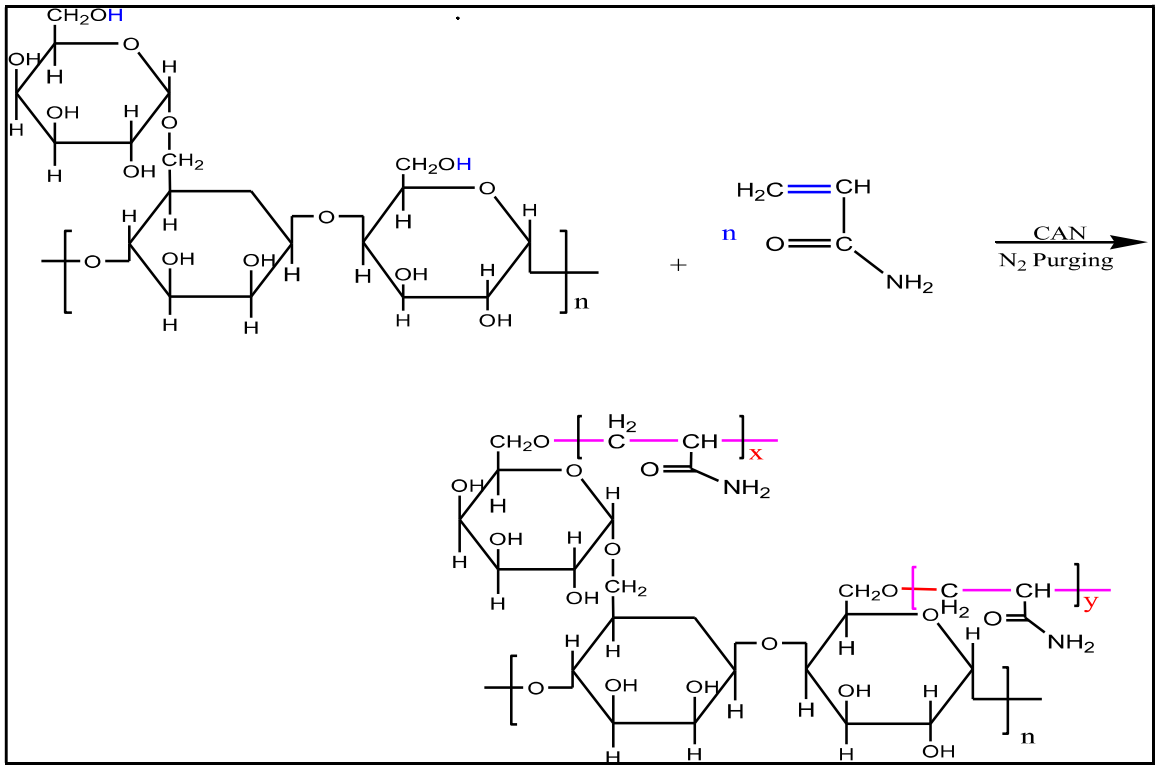


Figure 3.10 Polymerization reaction process of GG-g-PAM

3.5.6. Synthesis of Hydrolyzed Guar-gum grafted Polyacrylamide (H-GG-g-PAM)

The synthesis of H-GG-g-PAM was done by hydrolysis of GG-g-PAM with NaOH solution (Figure 3.11). The process of GG-g-PAM synthesis is given in previous section. 1 g of GG-g-PAM was dissolved in a 500 ml of distilled water in a beaker by electronic magnetic stirring at 50 °C. The reaction was continuously going on until GG-g-PAM dissolved properly. After that 20 ml of 1N NaOH solution was added. Then the reaction mixture was stirred using the magnetic stirrer for 3 h. Rest of the process is similar to H-PAM. Like H-PAM, H-GG-g-PAM was also partially hydrolyzed.

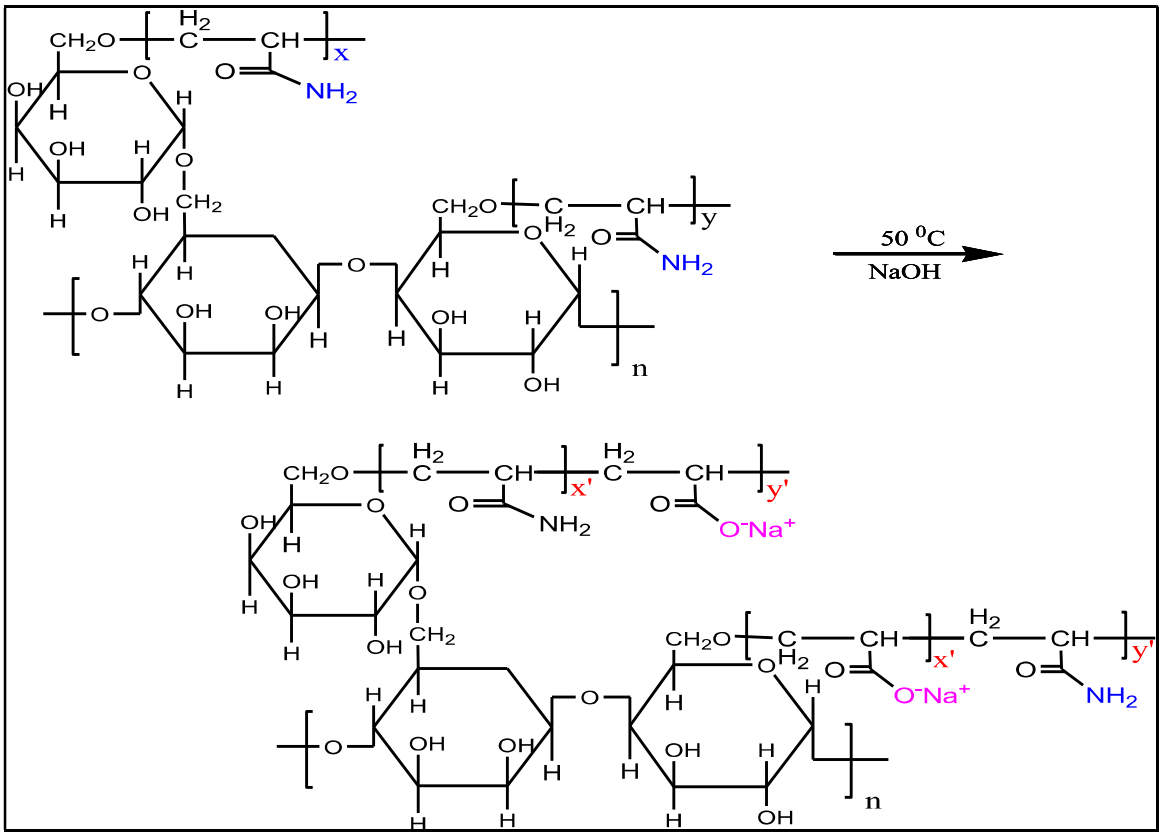


Figure 3.11 Polymerization reaction process of H-GG-g-PAM

3.5.7. Synthesis of Poly (2-acrylamido-2-methyl-1-propanesulfonic) acid-co-Polyacrylamide (PAMPS-co-PAM)

Copolymer of polyacrylamide and poly (2-acryamido-2-methylpropanesulfonic acid) (PAMPS-co-PAM) was synthesized by free radical polymerization technique using CAN initiator [91]. A schematic representation of polymerization process is shown in Figure 3.12. 2 g of AM was dissolved in 20 ml distilled water in a conical flask and stirring. Then 1 g of AMPS was dissolved in 100 ml distilled water and mixed with AM solution. The flask was placed in a water bath at 60 °C and N_2 gas was purged for 30 min. Rest of the process is similar to AP-g-PAM.

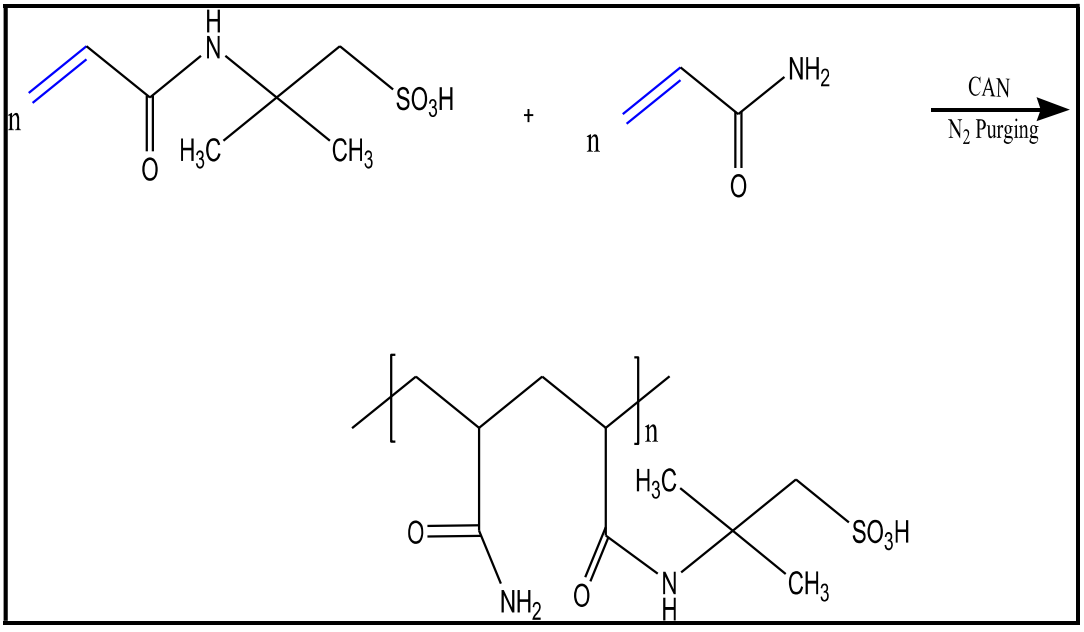


Figure 3.12 Polymerization reaction process of PAMPS-co-PAM

3.5.8. Hydrolyzed poly(2-acrylamido-2-methyl-1-propanesulfonic) acid - co-Polyacrylamide (H-PAMPS-co-PAM)

The synthesis of H-PAMPS-co-PAM was done by hydrolysis of PAMPS-co-PAM with NaOH solution (Figure 3.13). The complete process of PAMPS-co-PAM synthesis is already discussed in previous section. 0.5 g of PAMPS-co-PAM was dissolved in 1000 ml distilled water in a beaker of 2000 ml by electronic magnetic stirring at 50 °C. The reaction was going until the polymer dissolved properly. Rest of the process to obtain H-PAMPS-co-PAM is similar to H-GG-g-PAM.

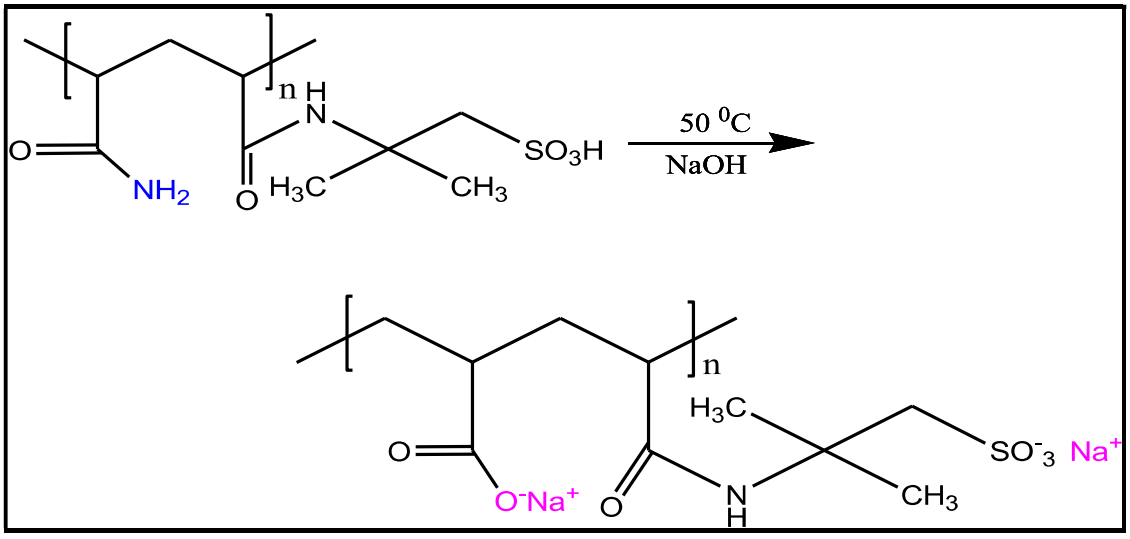


Figure 3.13 Polymerization reaction process of H-PAMPS-co-PAM

3.6 Characterization of Haul Road Dust

Size distribution of haul road dust sample was carried out by sieve analysis. Elemental compositions of dust were identified by energy dispersive X-ray (EDX) spectroscopy.

3.6.1 Size distribution

The typical design of a haul road is divided into four parts, mainly surface course, base course, sub-base and sub-grade from the top layer to bottom layer. Emission of dust largely depends on the particles size of the road surface course materials. According to D.S. Williams et al. (2008), fine particles of size below 100 μm have high tendency to get airborne and remain suspended in the air [36]. Above this size, rarely particles will be uplifted in the air from the surface of the unpaved road [40]. Smaller dust particles i.e. below 10 μm size will remain suspended in the atmosphere for long time and move up to 500 m or more from the source of generation. As per ASTM standard, particles below 2 mm size have been used to determine the moisture content of any soil sample with an accuracy of 99.99%. Sieve analysis of the dust sample is done using mesh sizes 10, 12, 20, 35, 50, 100 and 200 of ASTM standard [92].

3.6.1.1 Sieving analysis

At first, all the sieves and the pan are weighed separately. Then the sieves are placed in a stack of increasing size number which corresponds to decreasing opening size. Prepared sample of 500 g is poured into the top of the sieve stack and the cover is placed tightly on top. The sieve stack is placed into the sieve shaker and tightened by the clamp. Sieving is done for 10 min. Thereafter, all the sieves and the pan are weighted (along with the collected dust on them).

3.6.2 EDX spectroscopy

Elemental analysis of dust sample was carried out by EDX High Resolution (model 51N1000-EDS, Oxford Instruments Nano Analysis).

This is an analytical technique used for the chemical characterization or elemental analysis of a sample. It works on the fundamental principle that each element has a unique atomic structure allowing X-rays that are characteristic of an element's atomic structure to be identified uniquely from one another. It studies the interaction between a sample and a source of X-ray excitation. The x-axis of spectrum depicts the energy of the X-rays and y-axis represents the number of counts. The position of the peaks leads to the identification of the elements in the sample. The peak height helps in the quantification of each element present in the sample. The main advantage of EDX technique of characterization is that it can be used for both qualitative (type of elements) as well as quantitative (percentage of the concentration of each element) analysis of the sample. One more advantage of EDX analysis is that it is a non-destructive process, so it needs very little quantity of sample without any further processing.

3.7 Characterization of Synthesized Polymers

All the synthesized polymers were characterized by nuclear magnetic resonance of hydrogen atom (^{1}H NMR) spectroscopy, fourier transform infrared (FTIR) spectroscopy, thermo-gravimetric analysis (TGA) and intrinsic viscosity measurement.

3.7.1 ^{1}H NMR spectroscopy

^{1}H NMR analysis of the all the polymers were performed by NMR spectrometer (Model JEOL AL 500). The polymers were dissolved in D_2O. Tetramethylsilane (TMS) was used as an internal reference. The structure of the synthesized polymers can simply be identified by using ^{1}H-NMR spectra.

NMR is an analytical method used to define a unique structure of compound. There are many spectrometers available for structure analysis like H-NMR, C-NMR and N-NMR. Out of these, hydrogen (H-NMR) is the first and also the most common atom used in nuclear magnetic resonance spectroscopy.

Normally, the nuclear spins are random in direction. But when an external magnetic field is present, the nuclei align themselves either with or against the field of external magnet. The energy difference between the two spin states is measured and denoted by *chemical shift (δ).*

Chemical shift is a measure of how far the signal produced from the proton is away from the reference compound signal. The reference compound or TMS is at the zero positon on the very right of the spectrum, and as it moves toward the left, the ppm values become larger. It is the unit used to measure chemical shift. Normally, the proton chemical shifts come in the range of 0 to 15 ppm. The chemical shift is identical for a specific proton regardless of the spectrometer used.

The NMR spectrum is a plot between the intensity of NMR signal (y-axis) versus the magnetic field (frequency) (x-axis) in the reference to TMS. Number of separate signals in the NMR spectrum gives an idea that protons within a compound experience different magnetic environments. The position of signals in the spectrum gives an idea about how far

they are from the signal of the reference compound. This information tells the kind of proton or protons that are responsible for the signal.

3.7.2 FTIR spectroscopy

A Thermo Nicolet FTIR spectrophotometer (Model JASCO FT-IR-5300) was used to record the IR spectra of polymers within the range of 4000-400 cm^{-1}. Sample was mixed with KBr to form pellets which were used for this analysis.

FTIR instrument sends infrared radiation of 10000 to 100 cm^{-1} through the sample placed inside the sample chamber. There is an IR source from where IR radiation comes out and collides to a beam splitter to split the beam into two parts. One part of rays strikes to a stationary mirror and the other to a moving mirror. Both the rays return back and make intererance fringes. As both the parts of the rays have different wavelength, they may form coustructive wave or destructive wave. Now these waves move forwards and strike the sample. After striking, some of the wavelengths are absorbed by the sample and the remaining are transmitted. The transmitted waves are detected by the spectra detector. On the basis of this, the energy vs time spectra has been plotted, which is called as Inteferogram. Mathematical formula of fourier transform equation has been applied on it, to get the graph between frequency vs wave number. As each material is a unique combination of atoms, no two compounds produce exactly the same infrared spectrum. Therefore, infrared spectroscopy can result in a positive identification (qualitative analysis) of every different kind of material.

3.7.3 Thermogravimetric analysis (TGA)

A TGA-DTA (Elmer-Perkin STA 6000, USA) was used to record the TGA analysis of the synthesized polymers. TGA analysis of the sample is carried out under inert atmospheric condition at a temperature range from 30 °C to 500 °C with the heating rate of 10 °C per min.

Thermogravimetric analysis is a technique to identify the thermal behaviour of any substance. In this technique, the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. From TGA analysis, information come out mainly related with thermal stability of materials, fingerprint materials for identification & quality control, explain decomposition mechanism, oxidation of metals in air, corrosion studies, and estimation of product lifetime.

A graph is plotted in TGA, where the x-axis represents time or temperature and the y-axis, weight or weight percentage. As the temperature increases, material starts decomposition due to which weight of the substance decreases. This decomposition of material is due to the evaporation of volatile materials present in the polymer, breaking of chemical bonds, and reduction of polymer in ammonia or other substances.

3.7.4 Intrinsic viscosity

Viscosity measurements of the aqueous solutions of all polymers are carried out with the help of Ubbelohde Viscometer (P/2741). Intrinsic viscosity of all the samples is determined from

the point of intersection of two plots - extrapolated to zero concentration i.e., inherent viscosity (η_{inh}) versus concentration (C) and reduced viscosity (η_{red}) versus concentration (C).

Measurement procedure of intrinsic viscosity of polymers is described below. Polymer solution is prepared by dissolving 0.5 g of polymer in freshly prepared 100 ml distilled water with slow stirring. The volume of the stock solution is made up to 100 ml. From this stock solution, the polymer samples of various concentrations are prepared. The viscometer is introduced into a constant temperature bath (maintained at 25 ± 0.1 °C) and clamped to a stand to hold it vertically. About 25 ml of freshly prepared distilled water is introduced into the viscometer and kept in the bath for 20 min so as to allow the distilled water to come into thermal equilibrium with the bath. The distilled water was sucked into the upper bulb of the viscometer through the capillary and the time of flow in between the two marks is noted. Three consecutive readings are noted before taking an average value. This is noted as 't_0', the time of flow of distilled water (18 s). In the similar way, the time of flow of the polymer solutions at various concentrations is measured. The time of flow for the polymer solution is recorded as 't', for concentration 'C'. The reduced viscosity and inherent viscosity are calculated using the following relations:

Relative viscosity, $\eta_{rel} = t/t_0$ … (3.1)

Specific viscosity, $\eta_{sp} = \eta_{rel} - 1$ … (3.2)

Reduced viscosity, $\eta_{red} = \eta_{sp}/C$ … (3.3)

Inherent viscosity, $\eta_{inh} = \ln \eta_{rel}/C$ … (3.4)

3.7.5 Molecular weight measurement

Molecular weight of polymer can be estimated by using the Mark-Houwink equation, $[\eta] = KM^{\alpha}$, which is generally used for estimation of approximate molecular weight of linear polymers.

Here,

M = Molecular weight (Da),

η = Intrinsic viscosity ((dL/g),

K and α values are constants for a particular polymer/ solvent/ temperature system.

For PAM and PAM related polymers - other than hydrolyzed polymers - the values of K and α are 6.31×10^{-5} and 0.80 respectively.

3.7.6 Neutralization Equivalent

The objective of this test is to determine the amount of amide hydrolysis by neutralization equivalent calculation. In this experiment, it will standardize a solution of base using the analytical technique known as titration. As this reaction is basically quantitative, it is possible to determine the concentration of a base or acid in an aqueous solution with high accuracy. It helps to know whether the polymer is a hydrolyzed one or not. This also helps to know the extent the extent of hydrolysis that has taken place.

In order to determine when a solution has been exactly neutralized, an acid-base indicator is used that changes colour in a certain pH range. As the pH of a neutral solution is 7.0, an

indicator that changes the colour near this pH should be used for an acid-base titration. Phenolphthalein indicator changes colour in the pH range of 8.3 to 10.0 and can be used to determine when the correct amount of base has been added to an acidic solution to exactly neutralize it.

3.7.6.1 Titration Process

1. Initially, titration was done for standardization. 40 ml of 0.1 N HCL solution was titrated by 1 N NaOH solution.
2. 2 drops of phenolphthalein indicator was added to it.
3. 4.3 ml (V_1) of NaOH solution was added to neutralize HCL solution.
4. In the second titration, 0.2 g of hydrolyzed polymer was dissolved in 150 ml of distilled water.
5. 40 ml of 0.1 HCL solution was added to the polymer solution.
6. This reaction was allowed to go for 5 h.
7. 2 drops of phenolphthalein indicator was added to it.
8. Thereafter, NaOH (1 N) solution was added drop by drop until the colour had changed.
9. Volume of NaOH was measured in burette (V_2).

Neutralization equivalent (NE) was calculated as,

$$NE = x \times 1000/(y \times z) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3.5)$$

where,

x = Weight of polymer sample (0.2 g)

y = (V_1 - V_2)

z = 1N NaOH

3.8 Calculation for Polymer Dosage

Polymer dosage can be expressed either by solid content of the suspension or by suspension volume. For the present study, polymer dosage is expressed in terms of solid content of suspension i.e. weight percentage concentration. Calculation for a typical polymer dosage is given here.

Wt. % of 1000 ppm $= (1000/10^6) \times 100\ \%$

$= 10^{-3} \times 100\ \% = 10^{-1}\ \%$

$= 0.1\%$

The polymer dosage with respect to suspension volume was considered for studying the viscosity measurement. The polymer solution with respect to weight percentage was considered for studying the moisture retention efficiency of haul road dust sample. Stock solution of **0.1% (1000 ppm)** weight percentage concentration was prepared.

3.9 Laboratory Experimentation on Moisture Retention of Dust Samples

Experiments were carried out in the oven supplied by Associated Scientific Technologies, Delhi, India (ISO 9001-2000). Before starting the experiments, haul road dust sample was fully dried in the oven at 110 ± 5° C for 24 h. As per the ASTM Standard (D2216, 2010), 20 g dust sample of particle size minus 10 mesh was taken for calculation of moisture content [93].

Dust sample was spread on petri dish maintaining more or less a uniform layer, so that added solution also can spread uniformly (Figure: 3.14). After a few trials, it was found that 10 ml of polymer solution is optimum and make the dust layer completely wet leaving no excess solution runoff. Hence, during the whole study for retention of moisture content of dust, 10 ml of polymer solution was applied to 20 g of dust sample. The optimum concentration of 0.1% by weight solution was applied. The sample was weighed before and after spraying of the polymer solution. The treated sample was placed in the oven. Sample was weighed at each 1 h of intervals. Moisture retention efficiency of haul road dust with polymer treated solution was studied for 8 h duration. Each test was repeated 3 times and the average value was taken to improve the accuracy of measurements.

Similarly, the experiment was carried out using only distilled water, in place of polymer solution, and moisture retention efficiency of samples was calculated.

The study was conducted at three different temperatures, namely 25 °C, 35 °C and 45 °C. A few representative samples before and after heating are shown through Figures 3.15–3.18.

Figure 3.14 Dust sample without adding water or solution

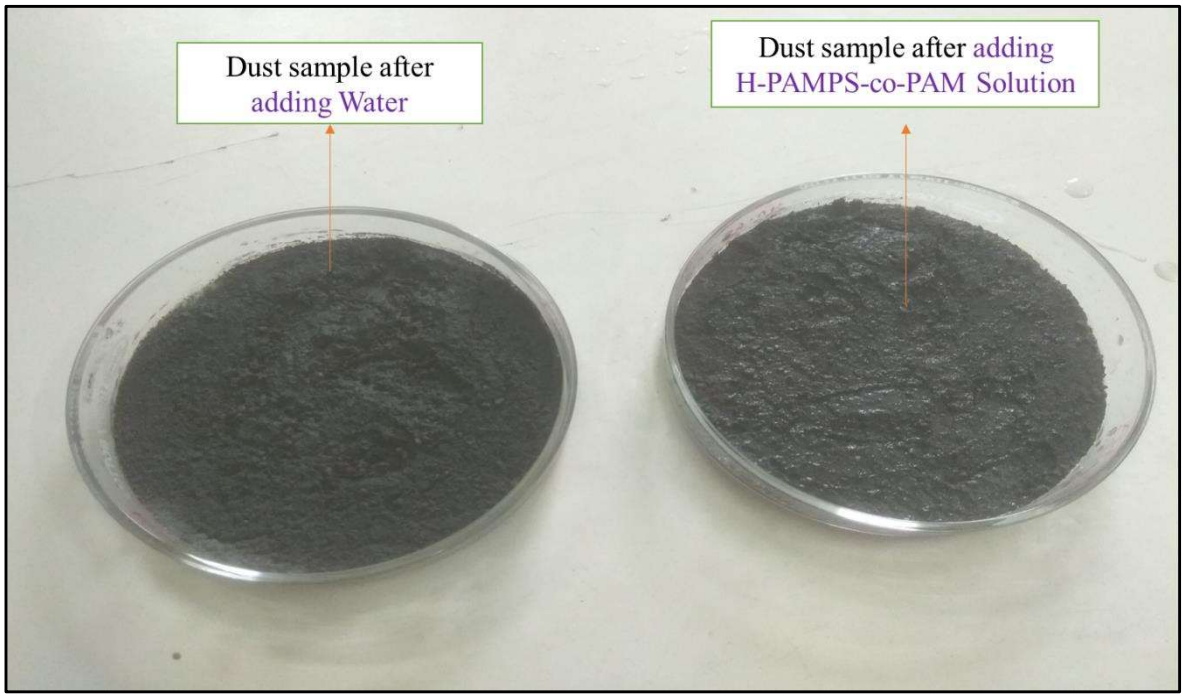


Figure 3.15 10 ml of water and H-PAMPS-co-PAM added to dust sample at 0th hour

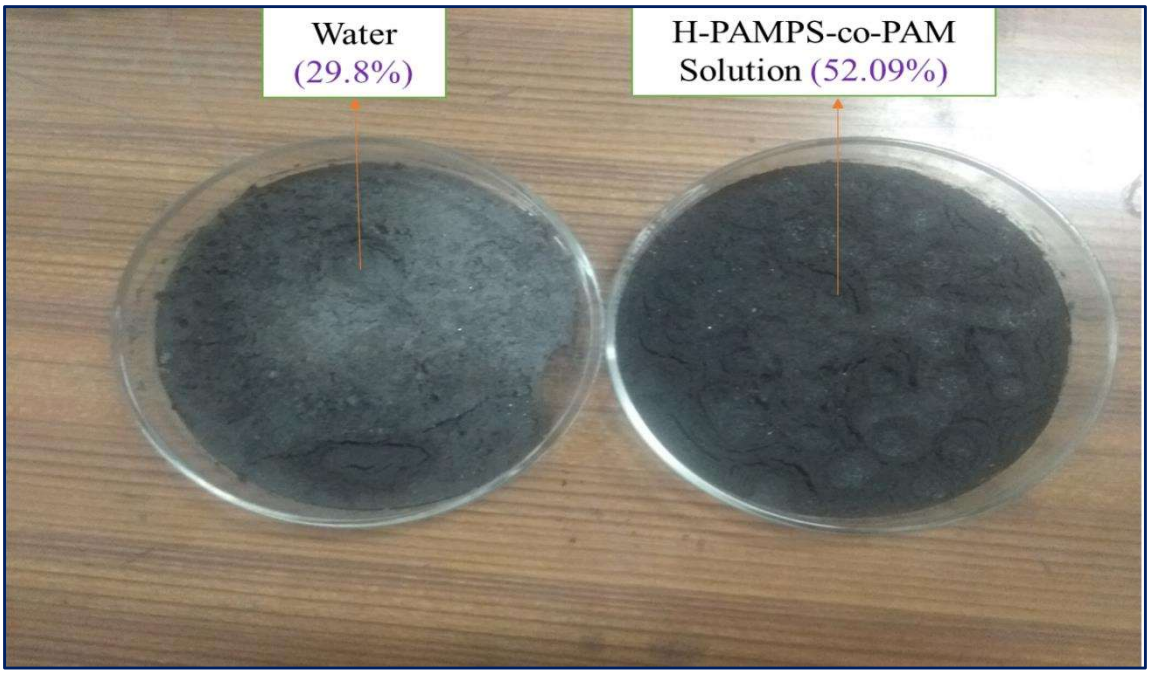


Figure 3.16 Water and H-PAMPS-co-PAM added on dust sample after 8 h in oven at 25 °C

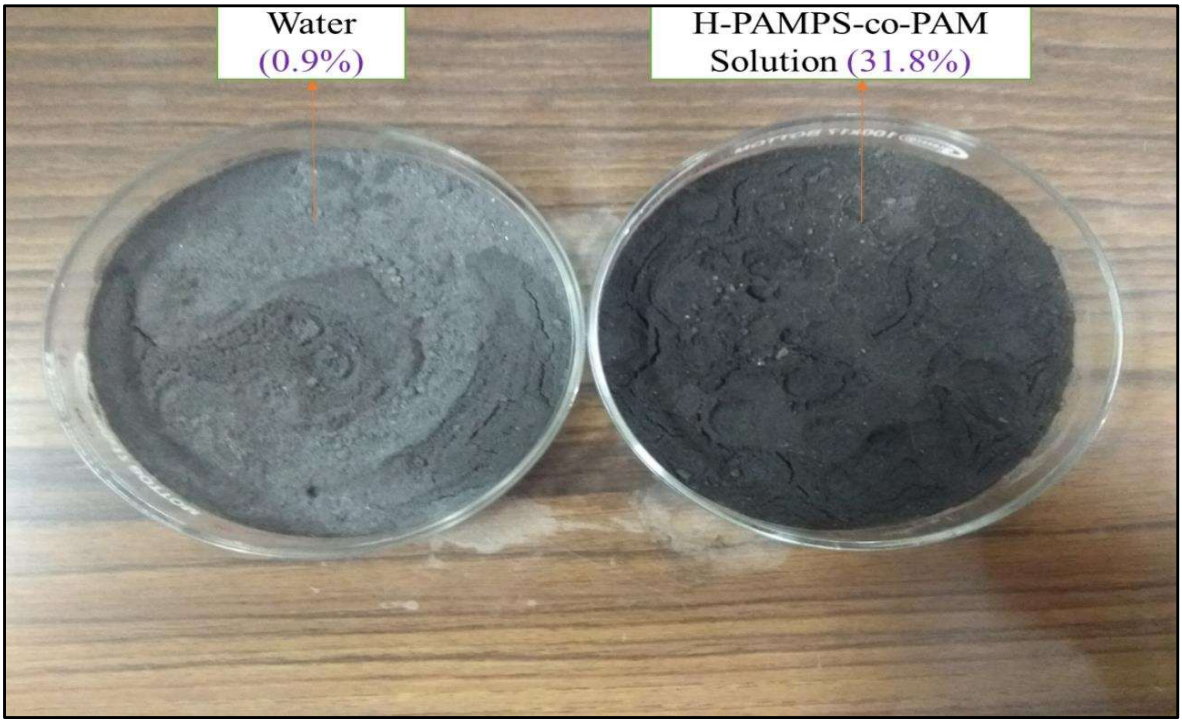


Figure 3.17 Water and H-PAMPS-co-PAM added on dust sample after 8 h in oven at 35 °C

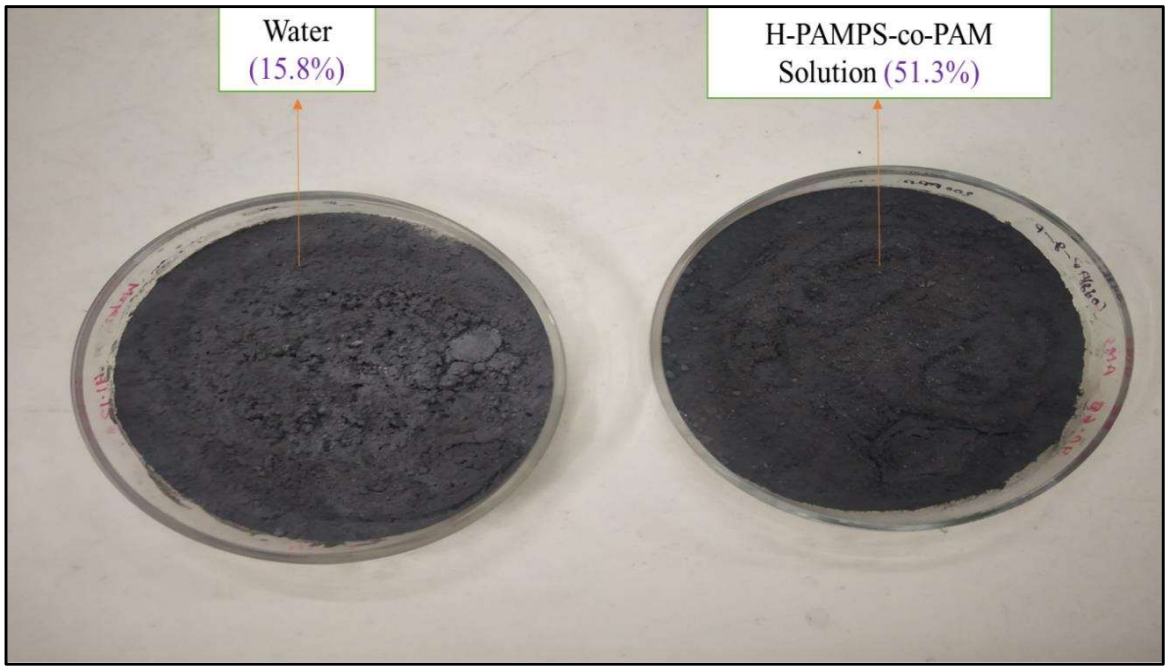


Figure 3.18 Water and H-PAMPS-co-PAM added on dust sample after 2 h in oven at 45 °C

3.10 Calculation for Moisture Retention Efficiency

Dust sample was placed in an oven at 110 °C for 120 h to drive out all the moisture content. Moisture content of dust sample was calculated as per the standards of ASTM D2216-2010. Calculation steps are given below.

W_P = Weight of petri dish

W_D = Weight of dust sample for the experiment (20 g)

W_1 = Weight of dust sample + Weight of petri dish

W_2 = Weight of dust + petri dish + polymer solution [Before putting inside the oven]

w (weight of polymer solution added) = $W_2 - W_1$

Now, polymer solution added dust sample (W_2) is put inside the oven and studied under three temperature conditions, namely, 25 °C, 35 °C and 45 °C. After 1 h of duration, measured its

weight (W_3) then further put inside the oven. Likewise weight was measured after each 1 h of duration up to 8 h.

W_3 = Weight of dust + petri dish + polymer solution [After 1 h of heating in the oven]

W_m (Weight loss of solution due to evaporation after 1 hr of application) = $W_3 - W_1$

M_L (%) (Percentage of moisture loss) = $[(W_m \times 100)/ w]$

M_R (%) (Percentage of moisture retention) = $100 - M_L$

Each test was repeated for 3 times to improve the accuracy. M_{R1}, M_{R2} and M_{R3} are the moisture retention percentage for three repeated tests. Finally, the average percentage of moisture retention is calculated and denoted by 'm' i.e.

$$m(\%) = (M_{R1} + M_{R2} + M_{R3})/3$$

This average percentage of moisture retention by dust sample is represented as moisture retention efficency.

3.11 Prediction of Haul Road Dust Emission Rate

USEPA is well known for carrying out experiment in the field of haul road dust emission and its mitigation. Industry knows very well that dust emission from haul road of opencast mine is about to 90% to 95% of total dust emission of a particular mine [94]. USEPA had started doing research on it from the early 1980's. It has proposed some empirical formula for predicting dust emission from haul road. Among these, AP42 report was published in the year of 2006. All the essence of the previous findings were merged into this, and gave the following empirical formulae to determine the dust emission rate.

For vehicles traveling on unpaved surfaces at industrial sites, emissions are estimated from the following equation:

$$E = k\,(s/12)^a\,(w/3)^b \quad \text{…….……(3.6)}$$

And, for vehicles traveling on publicly accessible roads, dominated by light duty vehicles, emissions may be estimated from the following equation:

$$E = [k\,(s/12)^a\,(S/30)^d / (M/0.5)^c\,] - C \quad \text{…….(3.7)}$$

Where,

k, a, b, c and d are empirical constants and

E = Size-specific emission factor (lb/VMT)

s = Surface material silt content (%)

W = Mean vehicle weight (tons)

M = Surface material moisture content (%)

S = Mean vehicle speed (mph)

C = Emission factor

A close look shows that equation (3.6) does not consider the effect of moisture content of the road surface material, which very important in the process of dust emission. Equation (3.7) appears to be more relevant than equation (3.6). But, equation (3.7) is applicable for light duty vehicles in publicly accessible roads.

Hence, both the above equations, proposed by USEPA, have their own limitations for being applicable to mine haul roads designed mainly for plying heavy vehicles.

There are some other mathematical equations available for determining the dust emission rate from haul road of mines (Table 3.4) (Patra, Gautam and Kumar, 2016) [40]**.** Like USEPA, other formulae also have their limitations. The empirical equation (3.8) given by

Chaulya et al. (2002) is claimed to be more suitable and accurate (90%) for Indian coal mining conditions [95]. As per this empirical equation, dust emission rate depends on six variables, namely moisture content, silt content, wind speed, average vehicle speed, frequency of vehicle movement, and capacity of the dumper.

Table 3.2 Empirical equations for estimation of dust emission rate from haul road

Authors	**Empirical equation**	**Variables**	**Remarks**
Chaulya et al. (2002) [95]	$E=[\{(100-m)/(m)\}^{0.8}$ $\{(s)/(100-s)\}^{0.1}\times u^{0.3}$ $\{2663+0.1(v+fc)\}\times 10^{-6}]$	m = moisture content (%) s = silt content (%) u = wind speed (m s^{-1}) f = frequency of loading (no. s^{-1}) v = average vehicle speed (m s^{-1}) c = capacity of dumpers (t) E = emission rate (g m^{-1} s^{-1})	Haul road in coal mines, SPM estimation
Chaulya (2006) [96]	$E=[(100-m)/(m)\}^{0.35}$ $\{(us)/(100-s)\}^{0.1}$ $\{0.5+0.1(f+0.42v)\}\times 10^{-3}]$	m= moisture content (%) s = silt content (%) u = wind speed (m s^{-1}) f = frequency (no. of holes d^{-1}) v = average vehicle speed (m s^{-1}) E = emission rate (g s^{-1})	Iron ore mines, TSP estimation
USEPA (1998) [97]	$E= 0.0031\ W^{3.5}$	W = Mean number of wheels E = emission rate (lb VMT^{-1})	Haul truck, TSP (<15 µm) estimation

Chakraborty et al. (2002) [98]	$E=[\{(100-m)/m\}\{s/(100-s)\}^{0.1}\times u^{1.6}\{1.64+0.1(v+f)\}\times 10^{-6}]$	m = moisture content (%) s = silt content (%) u = wind speed (m s^{-1}) f = frequency (no. of holes d^{-1}) v = average vehicle speed (m s^{-1}) E = emission rate (g s^{-1})	Coal/overburden, SPM estimation
Chaulya (2006) [96]	$E=[\{(100-m)/m)\}^{0.7}\{(us)/(100-s)\}^{0.1}\{41.6+0.03(fc+0108v)\}\times 10^{-5}]$	m= moisture content (%) s = silt content (%) u = wind speed (m s^{-1}) f = frequency (no. of holes d^{-1}) v = average vehicle speed (m s^{-1}) E = emission rate (g s^{-1})	Haul road in Iron ore mines, TSP estimation
Cowherd (1981b) [99]	$E= 0.0067W^{3.4}\ L^{0.2}$	W = mean no. of wheels L = surface silt loading (g m^{-2} E = emission rate (lb VMT^{-1})	Coal/overburden, TSP (<30 µm) estimation

$$E=\left[\left\{(100-m)/m\right\}^{0.8}\left\{s/\ (100-s)\right\}^{0.1}u^{0.3}\left\{2663+0.1(v\ +fc)\right\}\ 10^{-6}\right] \quad \text{.....(3.8)}$$

where,

m = Moisture content (%)

s = Silt content (%)

u = Wind Speed (m/s)

v = Average vehicle speed (m/s)

f = Frequency of vehicle speed (h^{-1})

c = Capacity of dumper (*T*)

E = Emission rate ($g\ s^{-1}\ m^{-1}$)

The value of silt content was found to be 20% through sieve analysis of the haul road dust sample. All other data except moisture and silt contents, were collected from the case study opencast coal mine as presented in Table 3.3. Emission rate was calculated for the moisture percentage after treating the dust samples with the polymer solutions, and again treating with water separately. When moisture content is below 1%, the emission rate is extremely high. Therefore, moisture content of dust below 1% is no more significant in the emission rate of haul road dust.

Table 3.3 Collected data for dust emission parameters of the case study mine

Silt content (s)	**Wind speed (u)**	**Avg. vehicle speed (v)**	**Vehicle movement frequency (f)**	**Vehicle capacity (c)**
20%	2.2 m/s	5.56 m/s	50 per h	60 T