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Application of Sulfonated GMA-g-non Woven PE Fabric for the Efficient Removal of Methylene Blue Dye from Wastewater

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Abstract: An environmental affable, economically feasible and reusable sulfonated adsorbent were constructed by gamma radiation grafting of glycidyl methacrylate (GMA) on non-woven polyethylene fabric and subsequent chemical modification. Highest graft yield of 343.31% was obtained at favourable conditions: 30 kGy radiation dose, 5% monomer concentration, adding up of 0.5% Tween-20 as an additive, 4 h reaction time. The epoxide group containing GMA-g-PE film were functionalized through sulfonation. The adsorbent was identified by Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM) and Thermo-gravimetric Analysis (TGA). The aqueous solutions of methylene blue were prepared in various concentrations and adsorption behavior by the developed sulfonated-GMA-g-PE film were investigated. MB uptake capacity at different environment such as contact time, pH and initial MB concentration were identified. The adsorption of MB is highly pH dependent and utmost sorption was found at pH 7. The kinetic adsorption data were interpreted by pseudo-first-order and pseudo-second-order equations. Pseudo-first-order rate kinetic model is more applicable for the sorption process due to its higher correlation coefficient. From the two isotherm model Langmuir and Fruendlich, Fruendlich model attuned best with the MB sorption as presented by higher correlation coefficient. The MB uptake capacity of the sorbent obtained from Langmuir model was 500 mg/g. Futhermore, the adsorbent could be reformed and reused repeatedly for the sorption of MB from waste water.

Keywords: Radiation, GMA, Methylene Blue, Isotherm, Kinetics

1. Introduction

Undesirable amounts of waste water containing color effluents are released from printing cosmetics, food coloring, dying, textile and papermaking industries [1]. Worldwide 10,000 commercial dyes are available and approximately 7×105 tones different types of dyes are formed each year [2]. Annually 12% of dyes produced are discharged as effluents from dying, textiles and related industries [3]. Even in small amounts of dyes revealing into water bodies can have a enormous repulsive consequence on the food web and aquatic life. It can create dermatitis, skin irritation and allergic reactions. Several of them are even mutagenic and

carcinogenic for human beings and aquatic organisms [4]. Alzheimer's disease in human body is also created by dyes [5]. On the other hand, it is seen that most of dyes are resistant to biodegradation and photo degradation [6–8]. So, it is very important to treat dye containing wastewater. Up to present time, several types of wastewater treatment techniques such as coagulation and flocculation methods [9], electro-coagulation [10] membrane separation [11], oxidation or ozonation [12, 13] and adsorption have been employed to eliminate dyes. Among these techniques, it is seen that for eliminating dyes and other pollutants adsorption is an efficient, lower cost, moderately simple and potentially useful technique [14, 15]. Several types of adsorbents

are developed that are used to eradicate dyes from waste water [16-23]. Due to comparatively lower adsorption capacity and secondary contamination of adsorbents many of them are not appropriate [24]. Uses of graft polymers has become new technique now. Elevated adsorption capacity and reusability of the adsorbent trim down the chance of creating secondary toxic waste. In case of grafting, monomers form covalent bonding to the polymer backbone. There are several advantages of polymer grafting. Grafted monomer can incorporate different functions with parent polymer but the mechanical property of the parent polymer is conserved [25, 26]. It is seen that over plasma treatment, decomposition of chemical initiators, oxidation of polymers etc, radiation grafting are more suitable as it has high penetration power to the polymer backbone and it forms mostly homogeneous free radicals very fast [27]. In the present study, sulfonated-GMA-g PE adsorbent was made by radiation induced grafting of GMA on PE and subsequent chemical treatment. The adsorbent can adsorb cationic dye methylene blue from aqueous solution. MB uptake capacity at different environment such as contact time, pH and initial MB concentration were identified. Kinetics and isotherm of MB adsorption were determined. Release of adsorbed MB and once more use of the adsorbent was also performed.

2. Experimental

2.1. Materials and Reagents

The non-woven polyethylene (PE) fabric that was obtained from Kurashiki MFG Co was used to prepare the adsorbent. Glycidyl methacrylate (GMA, CH₂=CCH₃COOCH₂CHOCH₂), Polyoxyethylene sorbitan monolaurate (Tween-20), Sodium sulphite, Sodium bisulphate were supplied by Sigma Aldrich (USA) and used without further purification. Hydrochloric acid (HCl), sodium hydroxide and Isopropyl alcohol (IPA) were purchased from Merck, Germany. Methylene Blue (MB) (Fluka, Switzerland), was used for the determination of MB uptake capacity of the adsorbent.

2.2. Preparation of Adsorbent

The preparation of adsorbent based on non-woven

polyethylene (PE) fabric were conducted in two steps. In the first step the PE fabric was cut into the size of 8 cm \times 2 cm and was irradiated by gamma radiation from Co-60 source. The radiation dose applied was 30 kGy at ambient temperature. Gamma irradiated PE film was kept in dry-ice temperature until use. At room temperature (25°C), 5 wt% monomer GMA and 0.5 wt% Tween-20 as emulsifier was added to deionised water to prepare the monomer GMA emulsion by stirring with a mechanical stirrer for 0.5 h. To remove dissolved oxygen the emulsion of GMA was bubbled for 40 minutes with Argon gas. The irradiated PE fabric was put into a glass bottle and it was fully filled by de-aerated GMA emulsion. To prevent inclusion of oxygen from air into the GMA-emulsion of bottle it was compactly closed with a lid. The grafting reaction was performed at 80°C in a water bath up to 4 h. The constructed GMA-g-PE film was washed repeatedly with deionized water to remove remaining monomer and homopolymer of GMA and dried up in a vacuum oven to steady weight. The degree of grafting was calculated as follows:

Degree of grafting (%)=(Wg - W0)/W0 \times 100

Where, Wg is the dry weight of GMA grafted PE fabric and W_0 is the dry weight of PE fabric.

In the second step, GMA-g-PE fabric was deeped in sodium bisulphite, sodium sulphite, iso-propanol and water solutions at the ratio of 10/3/10/77 wt% and the reaction was conceded at 80° C at constant heating up to 6h [28]. Subsequently, sulfonated-GMA-g-PE adsorbent was washed again and again with deionized water until neutral and desiccated in a vacuum oven to stable weight. The conversion of epoxide group (X) to sulfonate group were calculated as follows:

Conversion (X)=
$$\left[\frac{Ws-Wg}{104}, \frac{Wg-Wo}{142}\right] \times 100$$

Where, W_0 , Wg and Ws are the dry weight of PE fabric, GMA-g-PE fabric and sulfonated-GMA-PE adsorbent respectively. 142 and 104 are the molecular weight of GMA and sodium bisulphate. Figure 1 shows the preparation of sulfonate group containing MB adsorbent.



Figure 1. Preparation of sulfonated adsorbent from non-woven polyethylene for methylene blue adsorption from waste water.

2.3. Thermo Gravimetric Analysis

The thermal gravimetric analysis (TGA) under nitrogen environment of PE fabric and GMA-g-PE fabric sorbent (2 mg) were performed from 25°C to 480°C with a heating rate of 10°C/min from 25°C to 480°C and with a flow rate of 20 ml/min on a Perkin-Elmer TGA 7-thermal analyzer.

2.4. FTIR (ATR) Spectroscopy

We have characterized the PE and GMA-g-PE by FTIR (ATR) spectrophotometer (IRPrestige 21 supplied by Shimadzu Corporation, Kyoto, Japan) in the range 700–4000 cm^{-1} (resolution 4 cm⁻¹, number of scans: 20 times).

2.5. Scanning Electron Microscopy

The morphological experiment of the PE and GMA-g-PE febric was performed by using scanning electron microscopy (SEM) of Model JSM-6490LA, JEOL at an accelerating voltage of 20 kV. The SEM specimens were sputter coated with platinum.

2.6. Determination of MB Adsorption

The constructed sulfonated-GMA-g-PE films were immersed into aqueous solutions of MB at room temperature (25°C). Under different environment such as pH, contact time and initial MB concentration the adsorption was pursued. We have used HCl and NaOH solution for pH adjustment of the solutions. Aqueous MB solutions concentrations before and after sorption were determined by a UV spectro-photometer (wave length 663 nm). The MB dye sorption capacity of the constructed fabric was calculated using the subsequent equation:

$$Q = (C_1 - C_2)V/W$$

Where, Q is the sorption amount (mg/g of adsorbent), W is the weight of the sulfonated-GMA-g- PE fabric (g), V is the volume of solution (L), and C_1 and C_2 are the concentrations (mg/L) of dye before and after sorption, respectively.

2.7. Desorption of MB

MB dye desorption from the sorbent fabric was determined by soaking in 2M aqueous HNO_3 for 24 h. Percentage of desorption was calculated using the equation stated below:

Percent desorption={Ions desorbed (mg) /Ions adsorbed by the film (mg)}× 100

3. Results and Discussion

3.1. Preparation and Characterizations of the Adsorbent Film

3.1.1. Preparation of the Adsorbent Film

We have prepared sulfonated-GMA-g-PE adsorbent by gamma radiation (Pre-irradiation method). At first gamma irradiation was exposed to the non woven PE polymer backbone to form primary free radicals. Then, the monomer GMA solution mixing in a suitable solvent was added to the irradiated polymer for grafting. In the reaction graft growing chain and termination reaction was occurred. Finally the graft copolymer was produced. In pre-irradiation technique, monomer is not irradiated directly and formation of homo polymer is relatively lower than simultaneous irradiation technique. The GMA grafted PE film was functionalized by sulfonation reaction and the reaction was performed at 70°C, 80°C and 90°C at constant heating up to 6 h for the determination of effect of temperature on sulfonation. It was observed that comparatively homogeneous grafting was occurred at 80°C.

Grafting was carried out at 20kGy, 30kGy and 40kGy to study the effect of total dose of gamma radiation, maximum grafting and adsorption was found at 30 kGy radiation dose. 2.5%, 5% and 7.5% GMA solutions were used to optimize degree of grafting. Higher grafting was observed at 5% GMA solution. 343.31% graft yield was obtained (favourable conditions 30 kGy radiation dose, 5% monomer concentration, adding up of 0.5% Tween-20 as an additive, 4 h reaction time),

Epoxide group containing GMA-g-PE film can be easily functionalized. Using 343.31% degree of grafting, the sulfonation was performed in sodiumbisulphite, sodium sulphite and isopropanol and water solutions in the ratio of 10/3/10/77 wt% at 80°C at constant heating up to 6h. The constructed sulfonated GMA grafted films (G-343.31%) were modified by treatment with NaOH which increased the metal ion adsorption capacity of the films largely.

3.1.2. FTIR Analysis

FTIR spectrum of PE film and GMA grafted PE film were performed. IR spectrum of the PE film is shown in (Figure 2(a)). From IR spectrum of PE two types of vibrations, C–H stretching and C–H deformation vibrations were found. At 2910 cm⁻¹ and 2845 cm⁻¹ C–H asymmetric and symmetric stretching vibrations are seen. C–H bending, wagging and rocking deformation of CH₂ group are found at 1463, 1369 and 721 cm⁻¹ respectively.

Figure 2(b) shows the evidence for the grafting of GMA on PE film, peaks at 1716 cm⁻¹ indicates C=O stretching vibrations, peaks at 1273 and 1147 cm⁻¹ represents symmetric and asymmetric vibration of C-O in GMA and peaks for epoxy rings were appreared at 912 and 856 cm⁻¹.



Figure 2. IR spectrum of (a) PE fabric (b) GMA grafted non-woven PE fabric.



Figure 3. SEM image of (a) non-woven PE fabric (b) GMA grafted nonwoven PE fabric.

3.1.3. SEM Analysis of the Adsorbent

The SEM image of PE and GMA-g-PE fabrics are shown in Figure. 3. It was seen that the morphological structure changes from non woven PE to GMA grafted non woven PE and also physical appearance. In the SEM image, new graft chains and cross linked chains were found which provides proof of grafting.

3.1.4. Thermo-Gravimetric Analysis of the Adsorbent



Figure 4. TGA thermo-gram of (a) non-woven PE fabric (b) GMA grafted non-woven PE fabric.

The thermo-gravimetric analysis (TGA) for the non-woven PE film is presented in figure 4(a). Non-woven PE film shows thermal firmness up to 200°C and after 200°C it starts to degrade. It shows a three decomposition steps, i.e., 200°C–255°C, 255°C–499°C and above 499°C. At 200°C–255°C, 4.81% of the PE decomposes, at 255°C–379°C, 46.33% PE decomposes, at 379°C-499°C, 87.08% PE decomposes and at above 499°C, 93.38% PE decomposes.

Figure 4(b) represents the thermo-gravimetric analysis of the GMA grafted non-woven PE film. GMA grafted nonwoven PE film shows thermal firmness up to 180°C and higher than 180°C it starts to degrade. It shows a four decomposition state, i.e. at 180°C-184°C, 2.52% of the grafted PE decomposes, at 184°C-277°C, 60.36% grafted PE decomposes, at 277°C-358°C, 88.02% grafted PE decomposes and above 358°C, 97.38% grafted PE decomposes.

3.2. Investigation of MB Adsorption by the Sulfonated-GMA-g-PE adsorbent

3.2.1. MB Adsorption by GMA-g-PE Adsorbent

The prepared sulfonated-GMA-g-PE adsorbent is used for the uptake of MB from aqueous solution. Figure 5 shows the physical appearance of sulfonated-GMA-g-PE adsorbent before and after MB adsorption.



Figure 5. Sulfonated adsorbent before (a) and after (b) MB adsorption.

3.2.2. Proposed Adsorption Mechanism

Proposed adsorption mechanism for MB adsorption by sulfonated-GMA-g-PE is shown in Figure 6.



Figure 6. Proposed adsorption mechanism for MB adsorption by sulfonated-GMA-g-PE.

3.2.3. Effect of Adsorbent Weight

The effect of sulfonated-GMA-g-PE adsorbent weight on adsorption capacity of MB is shown in figure 7. It was seen that the MB uptake capacity of the adsorbent decreases with rising of adsorbent weight at the same volume and starting concentration of the MB solution. It is due to the reduction of the external area of the reaction site in a unit weight.



Figure 7. The effect of sulfonated-GMA-g-PE adsorbent on adsorption capacity of MB (conditions: Initial conc. 200ppm, pH 5.7, Volume 20 mL, Contact time 48h).

3.2.4. Effect of Contact time on Adsorption

The consequence of contact time on the adsorption amount of MB upto 72 h at initial pH 5.7 and the starting MB concentration 200 ppm for sulfonated-GMA-g-PE films were calculated. From Figure 8, it can be accomplished that the starting MB uptake rate is fast, then it slows down and gradually reaches to equilibrium at 48 h with the maximum adsorption of 73.85 mg/g. Almost 52% of the adsorption occurred within the first 5 hours. The early fast MB uptake rate is related to large initial concentration of MB and big large number of available active sites on sulfonated-GMA-g-PE adsorbent. This first MB uptake rate also indicates that most of the reactive adsorption sites are to be found in front of the surface of the sulfonated-GMA-g-PE adsorbent particle and easily attracted by MB.



Figure 8. Effect of contact time on MB adsorption by sulfonated-GMA-PE adsorbent (Conditions: Initial MB concentration200 mg/L, pH: 5.7; adsorbent 0.2606 g, volume 100 mL).

3.2.5. Kinetic Studies

Pseudo-first-order and pseudo-second order kinetic models were applied to know the kinetics and rate controlling steps

involved in the sorption process to test the experimental data collected from time dependent adsorption study at 25°C (Figure 8).

By the following equation pseudo-first-order kinetic model

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is expressed:

1

$$Log(Q_e-Q_t) = logQ_e - k_1/2:303 t$$

Where, Q_t and Q_e are the amount of MB adsorbed (mg/g) at any time and at equilibrium, respectively, k_1 is the rate constant (h⁻¹) of the first-order adsorption. The plot of log ($Q_e - Q_t$) against t would give the value of log Q_e from intercept.

By the following equation pseudo-second-order kinetic model is expressed:

Where k_2 (g/h.mg) is the rate constant of the pseudo second order adsorption. According to the pseudo -second order equation a plot of t/Q_t against t would give 1/Q_e from

 $t/Q_t = 1/k_2Qe^2 + t/Qe$

order equation a plot of t/Q_t against t would give $1/Q_e$ from the slope. Pseudo-first order kinetic plots is presented in Figure 9 and pseudo-second-order kinetic plots is shown in Figure 10. Pseudo-first order and pseudo-second-order kinetic parameters are shown in Table 1.

Table 1. The pseudo-first-order and pseudo-second-order rate constants for MB adsorption by sulfonated-GMA-g-PE adsorbent.

Qe (exp.) (mg/g)	Pseudo-first-order rate constant			Pseudo-second-order rate constant		
	Qe (theor.) (mg/g)	$K_1 (h^{-1})$	\mathbf{R}^2	Qe (theor.) (mg/g)	$k_2 (g h^{-1} mg^{-1})$	\mathbf{R}^2
73.85	69.66	0.278663	0.977	83.33	0.002057	0.947



Figure 10. Pseudo second-order plot for MB adsorption.

With respect to correlation coefficients R^2 the pseudo-firstorder model (0.977) gives better description of the MB adsorption on sulfonated-GMA-g-PE compared to pseudosecond-order kinetic model (0.943). It can also observed that the theoretical Q_e value (69.66) calculated from the pseudofirst order kinetic model (Figure 9) meets well with the investigated value (73.85) whereas theoretical Q_e value (83.33 mg/g) calculated from the pseudo-second-order kinetic model (Figure 10) is much higher than experimental value (73.85 mg/g). This implies that MB uptake capacity of sulfonated-GMA-g-PE pursued pseudo-first order rate process and the sorption process might have occurred by both physical and chemical adsorptions.



Figure 11. Effect of pH on MB adsorption (Initial MB concentration of 200 ppm and contact time of 24 h, adsorbent 0.04 g, volume 20 mL).

3.2.6. Effect of pH on MB Adsorption

There is excessive consequence of pH of the medium on sorption capacity of the grafted sorbent. At different pH values, because of protonation and deprotonation of sulfonate ions, the surface arrangement of the adsorbent could be exaggerated and could exist in dissimilar forms which can affect the sorption capacity. The effect of pH on MB adsorption at initial MB concentration 200 ppm and contact time 24h is shown in figure 11. It was seen that the MB uptake of the adsorbent increases from pH range 2 to 7. At pH=7 maximum adsorption of MB was observed. At low pH the adsorption capacity of the adsorbent is low, because of more protonation of sulfonate ions. With the increases of pH adsorption of MB increases, because of the deprotonation (forming SO₃ ions) of the modified sulfonated PE adsorbent which attracts the positively charged MB ions. At higher pH (more than 7) the adsorption capacity of the adsorbent decreases because at higher pH in basic media positively charged MB attracted by OH⁻ ions and then it is less attracted by negatively charged sulfonate ions.

3.2.7. Effect of Initial MB Concentration on Adsorption

Figure 12 shows the variation of MB uptake with the initial MB concentration (at initial pH 5.7 and time 48 h). It can be illustrated that with the raise of MB concentration, the adsorption amount of MB also raised. The reason behind this outcome is that the sites of chelation on the adsorbent surface become saturated when the concentration reaches higher value. When the chelating sites become occupied there remains no sites unoccupied for further sorption of dye.



Figure 12. Effect of initial MB concentrationon the adsorption capacity (pH 5.7 and time 48 h, adsorbent 0.022 g, volume 20 mL).

3.2.8. Adsorption Isotherms

The interactive relationship between adsorbent and adsorbate, surface homogeneity/heterogeneity of the adsorbent can be explained by adsorption isotherms. Langmuir and Fruendlich isotherm models are used to infer MB adsorption data.

The linear form of the Langmuir isotherm model is expressed by the following equation:

$$\frac{Ce}{Qe} = \frac{Ce}{Qm} + \frac{1}{Qmb}$$

Where, Ce is the equilibrium concentration (mg/L), Qm is the monolayer saturation adsorption capacity of the adsorbent (mg/g), Qe the equilibrium adsorption capacity (mg/L) and b the Langmuir adsorption constant (L/mg). Figure 13 shows the plot of C_e/Q_e versus C_e drawn from the investigational data shown in Figure 12. The relationship between C_e/Q_e and C_e is linear which indicates that the Langmuir adsorption isotherm is obeyed by the adsorption behaviour. The measured values of Q_m, b and R² obtained from the intercept and slope of the straight line are shown in table 2. From the straight line of the Langmuir isotherm model it is predicted that the adsorption data suits the Langmuir isotherm model as its correlation coefficient (R² 0.907) is close to unity. Overall, the Langmuir isotherm model can explain the adsorption of MB by the sulfonated-GMA-PE adsorbent. The utmost adsorption capacity of the adsorbent is 500 mg/g.

The separation factor (R_L) which is called dimensionless constant is an essential factor for Langmuir isotherm model. That describe the sorption character as favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$). From the Table 2 it is seen that R_L value is ($0.411 \sim 0.778$) which is less than unity which indicates that the MB uptake capacity by sulfonated-GMA-PE adsorbent is a favourable process.



Figure 13. Langmuir isotherm model for MB adsorption.

The Fruendlich isotherm model is expressed by the following equation:

$$\log Q = \log K_F + \frac{1}{n} \log C e$$

Where C_e is the equilibrium concentration (mgL⁻¹), Q_e is the equilibrium adsorption capacity, the sorption capacity (mgg⁻¹), and *n* is an empirical parameter. The plot of log Q_e versus log C_e shown in Figure 14. Figure 14 was drawn from the experimental data given in Figure 12. The K, *n*, and correlation coefficients (R²) values are given in Table 2.

Table 2. Langmuir and Freundlich isotherm parameters for MB adsorption.

Langmuir isoth	erm parame	ters	Freundlich isotherm parameters		
Qm (mg/g)	b (L/mg)	R ²	K _F (mg/g)	1/ n	R ²
500.0	0.0028	0.907	9.20	0.529	0.988

The data shown in the Table 2 indicates that there is a higher correlation coefficient (\mathbb{R}^2 , 0.988) for Freundlich model, hence it is more appropriate for recitation of the sorption of MB than Langmuir isotherm model (\mathbb{R}^2 , 0.907). This indicates that the sorption follows multilayer mode rather than monolayer mode. Which can describe that on the surface of the sorbent heterogeneous functional sites are strewn. The value of 1/n is less than unity (0.529) which demonstrates that Freundlich isotherm model is favorable for the adsorbent. Hence, it can be decided that Freundlich isotherm is more appropriate to explain the sorption of dye on the adsorbent.



Figure 14. Freundlich Isotherm model of MB adsorption on sulfonated-GMA-g-PE adsorbent.

The MB uptake capacity of the sorbent obtained from Langmuir model was 500 mg/g. The adsorption capacity of MB gained in the current study together with that reported in previous studies are presented in Table 3.

Table 3. MB sorption capacity of Sulfonated GMA grafted PE film compared with some other adsorbents.

Adsorbent	Adsorption capacity (mg/g)		
Sulphonated GMA grafted nonwoven PE	500		
(present study)			
Carbon monolith [29]	127		
Nanocrystalline cellulose [29]	101		
Activated carbons from walnut shells [30]	315		
Wheat shells [31]	21.50		
Gulmohar (Delonix regia) plant leaf [32]	186.22		
Tea waste [33]	85.16		
Thermally activated coir pith carbon [34]	5.87		
Jute fiber activated carbon [35]	225.64		
Acrylic acid-chitosan hydrogel [36]	322		

3.2.9. Regeneration and Reusability

In the current study, desorption of the adsorbent film was carried out by the usage of $2M \text{ HNO}_3$ for 24h at room temperature. So, the sorbent can be returned to the initial state and reused after MB adsorption.

4. Conclusions

An environmental affable, economically feasible and reusable sulfonated adsorbent were prepared by radiation grafting and subsequent chemical modification and has been considered for MB adsorption from aqueous solution. The adsorption study showed that the sulfonated-GMA-g-PE adsorbent can be used as a reusable adsorbent and it has high efficiency for MB adsorption. From the correlation coefficient of Langmuir and Freundlich adsorption isotherms, it was suggested that the Freundlich isotherm model is better to depict the sorption process. The experimental data for the adsorption of MB on the adsorbent fits well with the pseudo-first-order kinetic model based on the correlation between theoretical and experimental adsorption capacity. Experiment shows that the adsorbent can be reused for MB adsorption from waste water.

Abbreviations

GMA-glycidyl methacrylate, Polyethylene - PE, Methylene blue - MB

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