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Bulk Disposal of Unserviceable Toxic Cresylic Acid Waste Using Polymerisation Technique

P.K. Rai*, Amit Saxena, A.S. Rawat, Vivek Kumar, Mritunjay Pandit, and J.C. Kapoor

Centre for Fire, Explosive and Environment Safety, Delhi-110054, India
* *E-mail: pramrai@rediffmail.com*

ABSTRACT

A bulk amount of unserviceable toxic cresylic acid waste has been disposed off in a safe and environmentally benign manner. A process to immobilize this waste into non-toxic solid cresol formaldehyde polymer has been developed. Initial study was performed for 1.0 kg batch size for optimising the process parameters and conditions, and on the basis of this data, process was scaled-up for bulk disposal (100 kg/batch). The effect of ratio of reactants, type of catalyst [H_2SO_4, $NaOH$, and $Ca(OH)_2$], catalyst concentration, reaction temperature and reaction time have been studied in a batch process. Maximum immobilization in the 1.0 kg batch studies was obtained when cresylic acid and formaldehyde were taken in a molar ratio 1:1.5 using $NaOH$ as a catalyst. For bulk polymerisation, a ratio of 1.0 : 1.2 (cresylic acid : formaldehyde) with $NaOH$ [0.7 per cent (wt/wt of total charge)] was found optimum. The final polymerised product has been buried as per standard procedure in two brick lined pits. Finally, the site has been declared as free from the toxic waste.

Keywords: Cresylic acid, waste disposal method, polymerisation

1. INTRODUCTION

Cresylic acid is a generic term referring to three isomers of cresols methylphenol (C_7H_8O) or combination thereof. Cresylic acids are readily adsorbed through the skin and mucus membranes in liquid or vapour form and act as systemic toxins for which there is no established treatment. Literature has reported detailed study on toxicity, mechanism of action, skin/eye exposure, inhalation, carcinogenicity, first aid/clinical management, handling/storage and personal protection against cresylic acid[1]. Occupational health and safety administration (OSHA) have set 5 ppm or 22 mg/m^3 as permissible exposure limits (PEL's) for cresols. A huge amount of unserviceable cresylic acid waste was lying at one of our ministry of defence establishment since last three decades. The storage condition of cresylic acid waste, its toxicity and chemical reactivity made it a potential hazard for living things and, therefore, required immediate attention for its safe disposal in environmentally friendly manner.

There are biological[2-4] and chemical processes[5] for the destruction of cresylic acid. Anaerobical biological degradation of *o*-cresol by a microbial population has been suggested by Sulisti[2] , *et al.* and Mathur[3], *et al.* has discussed *p*-cresol biodegradation by using pseudomonas putida in batch reactor and in continuous bioreactor packed with calcium alginate beads. Biological processes are generally slow processes.

Polymerisation of *p*-cresol and formaldehyde is done by using a secondary amine (e.g., piperazine)[5]. Other techniques available for disposal of chemical wastes are incineration, derivatisation, and polymerisation. The first two methods are not suitable because of toxic gas evolution, formation of toxic intermediates, and limited applications. The storage condition and difficulty in handling of cresylic acid waste also disqualified incineration technique, hence polymerisation was considered.

The condensation polymerisation could be an important method of disposal of cresylic acid as phenol-formaldehyde resins are produced by the condensation polymerisation of phenols (phenol, cresol, resorcinol, etc.) and aldehydes (formaldehyde, acetaldehyde, furfuraldehyde, etc.) in the presence of catalyst (acidic or alkaline). The structure and properties of the polymer depends largely on to the molar ratio of the reactants and the nature of the catalyst used, i.e., novolak[5] (thermoplastic) and resol (thermosetting) resins are obtained from phenol and formaldehyde under different conditions. Gibson[7,8], *et al.* has synthesized the controlled molecular weight, linear, ortho- and para-cresol novolac oligomers. Miloshev[9], *et al.* discussed the polycondensation of *m*-cresol with formaldehyde catalysed by oxalic acid. He has also studied the influence of reagents molar ratio and the nature/quantity of the solvent used on the molecular weight characteristics of the oligomers formed. Chetan[10], *et al.* has synthesized *m*-cresol-formaldehyde ortho-novolac resins by varying the mole ratios of phenol or substituted phenol to formaldehyde. Curing kinetics of *o*-cresol formaldehyde epoxy resin has also been discussed[11,12].

Novolac resin is formed when an excess of phenol reacts with formaldehyde in molar ratio of 1: 0.6, in the presence of an acid catalyst such as HCl/H_2SO_4. It has a linear structure of macromolecules, and is a thermoplastic material with reversible fusibility and solubility. The lower the molar ratio of phenol to formaldehyde, the higher will be the molecular weight of the

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resin obtained. Increased duration of polycondensation results in combination of more phenol and formaldehyde molecules, and it results an increase in mean molecular weight of the end product. If *NaOH* is used as a catalyst with low ratio of phenol to formaldehyde then the polymer produced is resol type, i.e., thermosetting. Ishida[13], *et al.* while studying the formation of thermosetting resins via computer simulation reactions of phenols with formaldehyde also found opposite results with base-catalysed system *vs* acid-catalysed system. Water liberated in the process has only an extremely small effect on the equilibrium and the rate of the process. El-Hadi[14], *et al.* has discussed the thermodynamics of exchange of alkali-metal ions on variously cross-linked *m*-cresol formaldehyde resins. Bench-scale experiments as innovative treatment of cresols contaminated soil have also been performed[15].

Moreover, the conversion of organic waste into polymeric material depends on several factors such as chemical ratio, temperature, type and amount of catalyst and reaction time. Product nature and quality can also be altered by using base or acid medium. Over various disposal techniques, polymerisation has been considered for the bulk and safe disposal of unserviceable cresylic acid waste in environmentally friendly manner. Moreover, the objectives of the present study were to identify and quantify the waste, and to dispose off the waste using a suitable and safe disposal technique.

2. EXPERIMENT

2.1 Identification of Unknown Chemical

100 g of chemical waste was distilled at 195 ± 2 °C. Distillate was analysed using high performance liquid chromatography (HPLC) instrument from Perkin Elmer, Model 1022 equipped with sample injector, column used was C18 reversed phased (length of column = 25 cm and internal diameter = 4.6 mm). The pump used was 200 LC from Perkin Elmer, mobile phase was Methanol/water in the ratio of 40: 60 pumped at a flow rate of 1 mL/min, and the sample was detected at a wavelength of 218 nm. Prior to HPLC analysis, the distillate sample containing cresol was filtered through a 0.22 µm filter. For each injection 25 µm of sample was injected using a loop. Each standard/sample was run in triplicate. Peak area was used to calculate the concentration. Figure 1 represents the HPLC chromatogram.

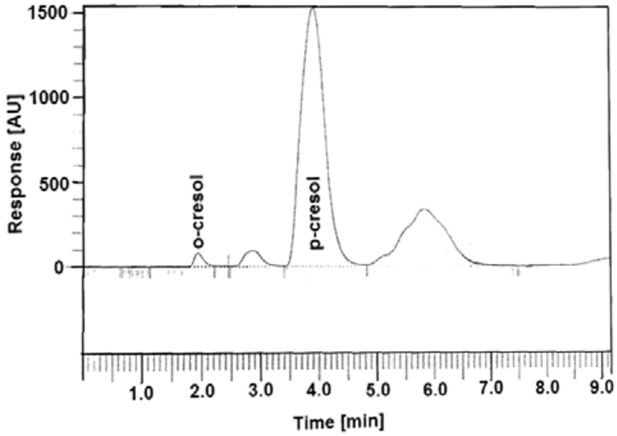

Figure 1. HPLC chromatogram of unserviceable cresylic acid.

The chemical was also analysed using gas chromatograph coupled with mass spectrometer (GC/MS) instrumental technique. GC/MS (EM 640 S) of Bruker, Germany was used for identification. It was equipped with HP-5 MS column of 30 m X 0.25 mm X 0.25 µ dimensions. Temperature programming [40 °C (2 min hold) to 280 °C (4 min hold) @ 10 °C/min] with split less injection technique was used to perform the study. Injection port and GC/MS interface, MS source and quadrupole analyser were kept at 250 °C, 150 °C, and 150 °C respectively. Figure 2 represents the GC/MS spectra of unserviceable cresylic acid and 4-methylphenol.

2.2 Disposal Technique (Polymerisation)

Polymerisation experiments were carried out with pure cresylic acid and collected sample with formaldehyde and catalysts (H_2SO_4, $Ca(OH)_2$ and $NaOH$) under different conditions (Table 1-4). For acid catalysed polymerisation, cresylic acid and formaldehyde was taken in a molar ratio of 1.0-1.6 : 1.0 along with 2.0 per cent (wt/wt of total charge) of H_2SO_4 in a three neck flask of capacity 500 mL. The flask was fitted with reflux condenser and stirrer. The content was then heated for 180 min at a temperature of 130 ± 2 °C. On heating the contents started getting viscous and separate out into two layers, the upper layer was that of water and the lower layer being that of liquid polymer. The reaction mixture was cooled and the polymer separates as semi solid which was decanted and washed repeatedly with water. Within 24 hrs the semi solid polymer became hard material. Reaction conditions and yields are presented in Table 1. Base catalysed polymerisation was performed using calcium hydroxide and sodium hydroxide separately. For calcium hydroxide [1.5 per

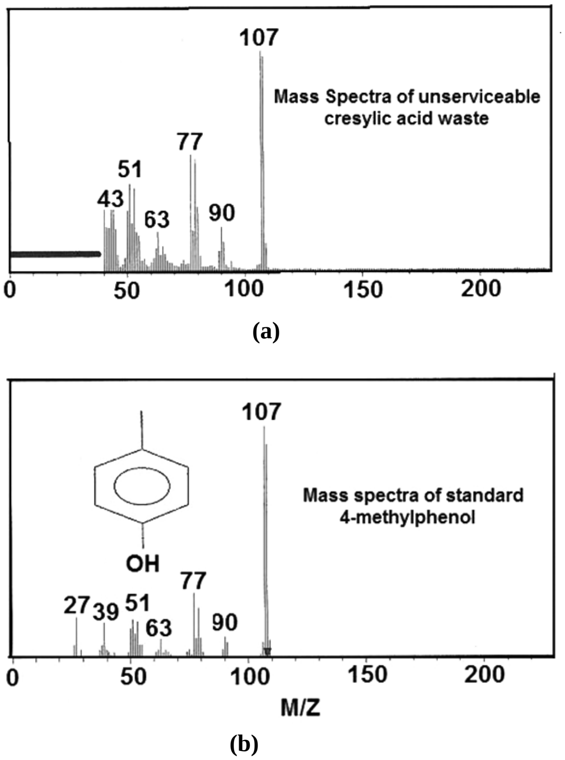

Figure 2. GC/MS spectra of unserviceable (a) cresylic acid and (b) 4-methylphenol.

cent (wt/wt of total charge)] catalysed polymerisation, cresylic acid and formaldehyde was taken in a molar ratio of 1.0 : 1.0-1.6. Reaction time and temperature were also varied from 50-60 min and 100-130 °C respectively (Table 2).

Table 1. Effect of molar ratio of reactants on sulphuric acid [2.0 per cent (wt/wt of total charge)] catalysed polymerisation

Molar ratio	Reflux time (min) / Temp (°C)	Yield (per cent)
1.0 : 1.0	180/130	93
1.2 : 1.0	180/130	97
1.3 : 1.0	180/130	95
1.6 : 1.0	180/130	88

Table 2. Effect of molar ratio of reactants on calcium hydroxide [1.5 per cent (wt/wt of total charge)] catalysed polymerisation

Molar ratio	Reflux time (min) / Temp (°C)	Yield (per cent)
1.0 : 1.0	60/100	78
1.0 : 1.2	60/100	83
1.0 : 1.4	60/100	88
1.0 : 1.5	60/100	96
1.0 : 1.6	60/100	95
1.0 : 1.5	55/120	96
1.0 : 1.5	50/130	97

For sodium hydroxide [2.4 per cent (wt/wt of total charge)] catalysed polymerisation, cresylic acid and formaldehyde were taken in a molar ratio similar to that with calcium hydroxide. Reactions were performed at 100 °C for 50 min (Table 3). The effect of temperature (70-130 °C) on sodium hydroxide catalysed polymerisation were also investigated (Table 4).

Table 3. Effect of molar ratio of reactants on sodium hydroxide [2.4 per cent (wt/wt of total charge)] catalysed polymerisation

Molar ratio	Reflux time (min) /Temp (°C)	Yield (per cent)
1.0 : 1.0	50/100	85
1.0 : 1.2	50/100	88
1.0 : 1.4	50/100	92
1.0 : 1.5	50/100	97
1.0 : 1.6	50/100	94

Table 4. Effect of temperature on *NaOH* catalysed polymerisation

Molar ratio	Temperature (°C)	Reflux time (min)	Yield (per cent)
1.0 : 1.5	70	70	93
1.0 : 1.5	90	60	95
1.0 : 1.5	100	50	97
1.0 : 1.5	120	45	98
1.0 : 1.5	130	40	98

2.3 Bulk Polymerisation at 100 kg/Batch Plant

Based on the data generated at lab-scale design (1.0 kg batch size) a bulk disposal process of capacity 100 kg/ batch, incorporating all safety measures was developed for the polymerisation of unserviceable cresylic acid to non-fusible and non-soluble thermosetting plastic followed by burial of waste polymer into pits. The schematic of plant has been given in Fig. 3. The performance of every components of plant was evaluated and then, the trial runs with 30 and 50 L batch capacities were carried out initially. Thereafter, five trials of 100 L batch size were conducted for the optimisation of the process parameters.

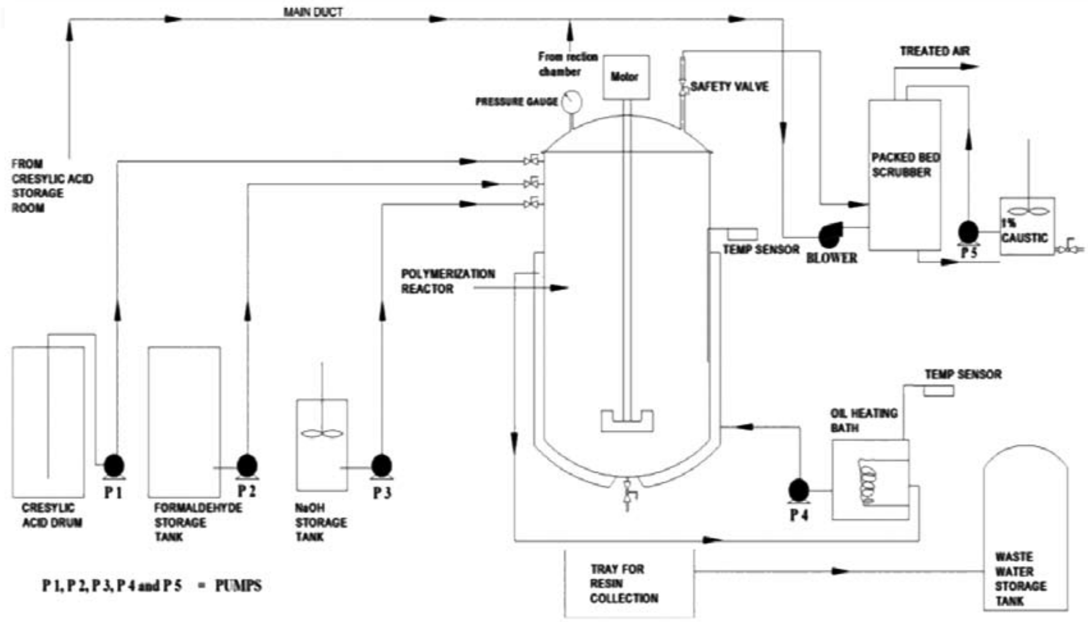

Figure 3. Schematic of disposal of cresylic acid (100 kg/batch).

Finally, cresylic acid and formaldehyde were taken in a molar ratio of 1.0: 1.2 with sodium hydroxide [0.7 per cent (wt/wt of total charge)] as a catalyst. The reactants (100 L cresylic acid, 90 L formaldehyde and 7 L of 20 per cent *NaOH* in water) were transferred into the polymerisation reactor using leak proof metering pumps (P1, P2 and P3). The polymerisation reaction would take placed at 90 ± 5 °C into a steel vessel jacket in 50 min. The reaction temperature was maintained by circulating pre heated oil (using pump P4) into the jacket of the reactor. When the reaction was completed, the vent line of the reactor is opened so that gases go to the main suction line, which finally goes to a scrubber system having 1.0 per cent *NaOH* aqueous solution sprayed using pump P5 to remove unreacted chemicals. The product is taken out from the bottom outlet into stainless steel trays.

Leakage test was carried out during the plant operation. The vent of the reactor and chemical storage rooms was connected to a high powered blower (5000 CFM) and the air was sent through a caustic scrubber system to remove any cresylic acid vapour from the air before discharging it to atmosphere. Leak proof ducting line was provided in the whole plant.

2.4 TGA/DSC Study of Bulk Polymerised Cresylic Acid

Thermograms for materials were recorded from 44 to 1020 ˚C in nitrogen using thermo gravimetric analyser, TGA/DSC from Perkin Elmer, USA. The initial sample weight was 10 mg and the heating rate was 20 °C/min. Figure 4 shows the TGA/DSC profile.

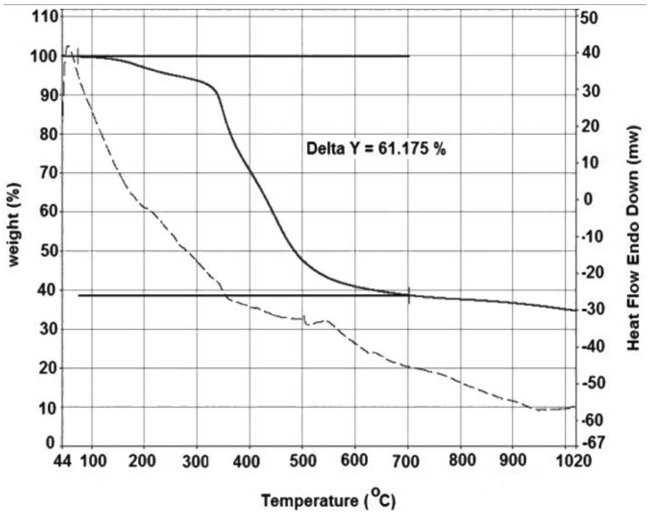

Figure 4. TGA/DSC of polymerised product of unserviceable cresylic acid.

3. RESULTS AND DISCUSSION

Unserviceable chemical waste was brownish in colour with pungent phenolic odour and acidic in nature (*pH* = 3.2). Distilled fraction collected at 195 ± 2 °C was yellow in colour and weighed, approximately 70 g. HPLC chromatogram indicated that cresols got separated within 3.85 min as shown in Fig. 1. The amount of cresol present in unknown sample was found to be 85 ± 1.0 per cent. HPLC analysis indicated the unknown sample to be cresylic acid. GC/MS characterisation of unknown chemical (compared with standard, Fig. 2) also indicated it to be ortho- and para-cresol (cresylic acid). On the basis of HPLC & GC/MS results and physico-chemical properties (density, boiling point, etc.) the chemical was identified as a mixture of ortho- cresol (2-methylphenol) and para-cresol (4-methylphenol) with 85 ± 1.0 per cent purity.

Results of sulphuric acid catalysed polymerisation of cresylic acid are presented in Table 1. At a molar ratio of cresylic acid : formaldehyde = 1.0 : 1.0 the yield (solid polymer) was 93 per cent. When the ratio of cresol : formaldehyde was increased to 1.2 : 1.0 yield increased to 97 per cent. Further, increase in cresol result a decrease in yield. The structure and properties of the polymer synthesized depends chiefly on to the molar ratio of the reactants and the nature of the catalyst used. Sulphuric acid catalysed polymerisation with low concentration of formaldehyde resulted low molecular weight, thermo plastic branched polymer, called novolac resin[5-10]. Novolac has linear structure of macromolecules with reversible fusibility and solubility. Reversible fusibility and solubility of novolac resins makes it a non acceptable disposed waste. Therefore, the polymerisation of unserviceable cresylic acid to novolac (a thermoplastic) could not be the best method for waste disposal. If unserviceable cresylic acid is converted to non-fusible and non-soluble thermosetting plastic then it could be the accepted way of disposal of waste. Ishida[13], *et al.* while studying the formation of thermosetting resins via computer simulation reactions of phenols with formaldehyde found opposite results with base-catalysed system *vs* acid-catalysed system. Therefore, the polymerisation was performed using base (calcium hydroxide and sodium hydroxide) catalysed reactions.

Cross-linked thermosetting resins are produced via calcium hydroxide [1.5 per cent (wt/wt of total charge)] catalysed polymerisation of unserviceable cresylic acid (Table 2). The polymerisation at a molar ratio of cresylic acid : formaldehyde :: 1.0 : 1.0 at 100 °C resulted 78 per cent yield in 60 min. When the ratio of cresol: formaldehyde was decreased to 1.0:1.2 yield increased to 83 per cent. Further, decrease in cresol ratio the polymerisation result an increase in yield (96 per cent, Table 2). When the temperature of the reaction was increased from 100 °C to 120 °C and 130 °C the yield was almost same but the reaction time decreased to 5 and 10 min respectively. When cresylic acid, formaldehyde and base catalyst are mixed in desired amount then initially hydroxymethyl phenols are formed. This results in a thick reddish brown resin. Reactive species is phenolic anion formed by deprotonation of acid. Negative charge is delocalised over aromatic ring which then reacts with formaldehyde. The reaction rate law that applies to this reaction is not a simple one but a highly complex one. Under these conditions a polymer with a linear structure is formed. On further heating methylene and ether bridges are formed, at this point resin starts cross-linking and transforms into a three dimensional polymer called resite. It is an infusible and insoluble material, and called thermosetting plastics. This highly cross-linked structure gave hardness and thermal stability to the final product and makes it impervious to chemical attack and salvation.

Table 3 represents the results of sodium hydroxide [2.4

per cent (wt/wt of total charge)] catalysed polymerisation of cresylic acid. Sodium hydroxide catalysed polymerisation at a molar ratio of cresylic acid : formaldehyde :: 1.0 : 1.0 at 100 °C resulted 85 per cent yield in 50 min, where as calcium hydroxide with similar ration of reactants resulted 78 per cent yield in 60 min (Table 2). This indicated that sodium hydroxide is a better catalyst than calcium hydroxide. Further increase in formaldehyde concentrations to 1.5 times the yield increased from 85 per cent to 97 per cent. In addition to that cresylic acid to formaldehyde in a ratio of 1.0 : 1.6 showed lesser yield than a ratio of 1.0 : 1.5 with both calcium and sodium hydroxide catalysed polymerisation. In order to optimise the polymerisation process for maximum yield in minimum time the temperature of the reaction mixture was varied from 70 °C to 130 °C (Table 4). Results indicated that as the reaction temperature increases the reaction time decreases. At 70 °C the reaction took 70 min to yield 93 per cent. As the reaction was performed at 100 °C the yield increased to 97 per cent and reaction time decreased to 50 min. Reaction time further decreased to 40 min with 98 per cent yield at 130 °C.

The polymer produced was tested for its plasticity by heating it in oven; it hardened on heating and behaved like Bakelite. This indicated the polymer to be thermosetting. Similar type of results was obtained with sodium hydroxide catalysed polymerisation. Therefore, If base is used as a catalyst with low ratio of cresol to formaldehyde then the polymer produced is resol type, i.e., thermosetting. Water liberated in the process has only an extremely small effect on the equilibrium and the rate of the process, and the basic factors which determine the direction of the reaction are temperature, duration of the reaction and the nature of the catalyst used. Results indicated that the base catalysed polymerisation not only produces thermosetting product but also requires much lesser time than that of acid catalysed polymerisation (Tables 1 and 4). Moreover, the study clearly indicated the suitability of base catalysed polymerisation for the disposal of unserviceable cresylic acid.

Based on the data generated at lab-scale design (1.0 kg batch size) a bulk disposal process of capacity 100 kg/ batch was developed and cresylic acid was polymerised. As per finally proposed methodology cresylic acid and formaldehyde were taken in a molar ratio of 1.0 : 1.2 with sodium hydroxide [0.7 per cent (wt/wt of total charge)] as a catalyst. Whereas, lab scale (1 kg/batch) study (Table 1) indicated the best ratio to be, cresylic acid : formaldehyde :: 1.0 : 1.5 and sodium hydroxide [2.4 per cent (wt/wt of total charge)]. This clearly indicated that at bulk process the requirement of formaldehyde and catalyst sodium hydroxide is reduced. The reduced requirement of reactants can be attributed to reduced evaporation (wastage) of formaldehyde during bulk polymerisation due to the completely closed reactor. The reaction temperature for bulk polymerisation also reduced from 100 °C to 90 °C. Moreover, in bulk process the three-dimensional availability of reactants with each other is increased, hence, the reaction proceeded faster.

Finally, polymerised solid cake was tested for leaching of un-reacted cresylic acid before buried into the pits. HPLC results indicated that the leaching of un-reacted cresylic acid was less than 5 ppm. Resol polymer is a cross-linked thermoplastic polymer, hence, no reversible reaction is possible and it remains stable for longer duration. Resol polymer is not degraded to any toxic products. After long duration of time, possibly, it may react with available atmospheric oxygen to corresponding acids and finally to carbon dioxide and water. Moreover, the polymer buried in pits will not cause any problem to future environmental safety.

TGA studies of polymerised cresylic acid (Fig. 4) waste in nitrogen atmosphere indicated that the polymer losses only 7 per cent of its weight up to 330 °C, which is caused by the evaporation of water and small molecules. The weight loss from 330 °C to 550 °C is approximately 49 per cent, which may be due to the breakage of ether linkages and carbonyl groups[16]. Thereafter, the polymer is almost stable and up to 1020 °C.

Moreover, the plant was utilised successfully with the capacity of five batches (100 kg/batch) per day. Unserviceable cresylic acid waste has been successfully disposed off by converting it into non toxic solid cresol formaldehyde polymer in a safe and environmentally benign manner. The final polymerised product has been buried as per standard procedure in two brick lined pits and finally, the site has been declared as free from the toxic waste.

4. CONCLUSIONS

Huge amount of toxic unserviceable cresylic acid waste was lying under unsafe storage conditions at one of our ministry of defence establishment since last three decades. This posed a serious hazard and needed safe disposal in an environmentally benign manner. The chemical was first identified and then analysed for its purity using HPLC and GC/MS techniques. The chemical was found to be ortho- and para-cresol and purity of the sample was 85 ± 1.0 per cent. A polymerisation process (polymerisation of the cresylic acid using phenol-formaldehyde polymerisation process) for the safe disposal was developed. The effect of ratio of reactants & catalyst, type of catalyst [$NaOH$, H_2SO_4 and $Ca(OH)_2$], reaction temperature and reaction time were studied to get the maximum polymerisation in minimum time and at lowest possible temperature. At a molar ratio of cresylic acid : formaldehyde = 1.2 : 1.0 the yield (solid polymer) was 97 per cent with sulphuric acid catalysed polymerisation. Overall, NaOH was found to be the best catalyst for polymerisation of cresylic acid. The study indicated the best ratio to be 1 : 1.5 : (cresylic acid : formaldehyde) with NaOH catalysis. At this ratio the yield was 98 per cent within 40 min at 130 °C. Moreover, results indicated that the base catalysed polymerisation not only produces thermosetting product but also requires much lesser time (45 min) than that of acid catalysed polymerisation (180 min).

Initially, the study was performed for 1.0 kg batch sizes and on the basis of it the bulk disposal process (100 kg/batch) had been completed. The bulk disposal of cresylic acid waste into non toxic solid cresol formaldehyde polymer was carried out successfully in an environmentally friendly manner. The final polymerised product has been buried as per standard procedure in two brick lined pits and finally, the site has been declared as free from the toxic waste.

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Contributors

Dr Pramod Kumar Rai obtained PhD (Chemistry) from University of Rajasthan, Jaipur in 1992. Currently working as Scientist ‘E’ at Centre for Fire, Explosive and Environment Safety (CFEES), Delhi. He is working in the field of chelating polymers, carbons, fire retardants, waste disposal techniques, nano-materials and adsorption science. His scientific contribution involves: Development of pitch-based activated carbon spheres, ABC fire extinguishing powder, fire suppressing gel compositions, and metal oxide nanoparticles-based adsorbents. He is honored with *DRDO Spin-off Technology Award* in 2006.

Dr Amit Saxena obtained PhD (Chemistry) (Nanomaterials) from Jiwaji University, Gwalior, in 2009. Presently working as Scientist ‘C’ at CFEES, Delhi. He is working in the field of NBC defence especially protection against toxic chemicals, nanomaterials, adsorption science, fire retardants and waste disposal techniques. His scientific contribution involves: Development of adsorbents for CO filters, CO canister, Mark-II TCD paper, ABC fire extinguishing powder, fire suppressing gel compositions, and metal oxide nanoparticles-based adsorbents. Technology of ABC powder has been transferred to private firm.

Mr Ashok Singh Rawat received MSc (Chemistry) from Institution of Chemist, Kolkata in 1995. Presently working as TO ‘A’, at CFEES, Delhi. He is working in the field of analytical chemistry and waste disposal techniques. His scientific contribution involves : Development of chelating polymers, pitch-based activated carbon spheres, ABC fire extinguishing powder, fire suppressing gel compositions, and metal oxide nanoparticles-based adsorbents. He is honored with *DRDO Spin-off Technology Award* in 2006.

Mr Vivek Kumar did BSc in 1992 from Delhi University, Delhi. Presently working as TO ‘A’, at CFEES, Delhi. He has expertise of working in the field of analytical chemistry and waste disposal techniques. His scientific contribution involves: Development of pitch-based activated carbon spheres, ABC fire extinguishing powder, fire suppressing gel compositions, and metal oxide nanoparticles-based adsorbents. He is honored with *DRDO Spin-off Technology Award* in 2006.

Dr Mritunjay Pandit received his PhD (Chemical Engg.) from IIT Delhi in 1997. Presently working as Scientist at CFEES, Delhi. He is working for management of hazardous wastes. He is honored with *DRDO Spin-off Technology Award* in 2006. He has filed one patent. He is a co-author of one chapter in a book. He has guided three PhD students and several MTech and MSc projects.

Mr Jagdish Chander Kapoor received MSc from Bombay University in 1980. He retired as Director, CFEES, Delhi in 2010. Many DRDO projects of strategic importance have been completed in his guidance. He has published/presented several papers in national and international journals/conferences and filed many patents.