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Conversion of Waste Polystyrene to Fuel Oil using High Alumina Cement as Catalyst

H. A. Deepa*, N. V. Chandini , Himanshu Mishra, Priyanka Sahu, R. Neelesh, R. Ravishankar and G. N. Vikas

Department of Chemical Engineering, Dayananda Sagar College of Engineering, Bengaluru - 560111, Karnataka, India; deepaha@gmail.com

Abstract

The global issue of plastic waste accumulation requires a scientific approach to transform it into valuable resources like fuel oil, thereby reducing environmental pollution. Waste polystyrene plastic was converted into fuel oil through catalytic pyrolysis, focusing specifically on the utilization of high alumina cement as a catalyst. A batch reactor of 549.77 cm^3 capacity was fabricated for catalytic pyrolysis. The surface properties of high alumina cement were investigated using Brunauer-Emmett-Teller (BET) analysis and compared with those of natural zeolite. The resulting pyrolysis fuel oil was characterized using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups, and Gas Chromatography-Mass Spectrometry (GC-MS) to determine its components. The findings contribute to the understanding of the efficacy of high alumina cement as a catalyst in waste plastic pyrolysis and its potential advantages over traditional catalysts.

Major Findings: *Reduction of polystyrene waste entering landfills, thereby contributing to the overall decrease in solid waste accumulation. Identification of novel catalyst with better surface area for catalytic pyrolysis of waste polystyrene. Fabrication of a batch reactor to improve the yield and reduce the time taken for catalytic pyrolysis of waste plastic*

Keywords: *Batch Reactor, Catalytic Pyrolysis, FTIR Analysis, Fuel Oil, GC-MS Analysis, High Alumina Cement, Waste Polystyrene*

1.0 Introduction

Plastics are integral to everyday life because of their light weight, low cost, water resistance, malleability and robustness[1,2]. Polystyrene (PS) is among the most popular plastics, valued for its strength and durability[3]. PS finds applications in building insulation, containers for food, plates, and packaging foam[1-4].

However, because majority of the plastic products are designed for one-time use, the amount of plastic waste is steadily increasing, leading to significant environmental pollution. This "white pollution" has sparked widespread concern about reducing and recycling plastic waste. Since plastics have low density but high volume, relying on landfills for disposal consumes valuable land resources. Additionally, there is a hazard of pollutant discharge with microplastic debris found in subsurface water and soil[1]. Incinerating plastics to recover heat, steam and electricity is another common disposal method, but it can release harmful fumes, including polycyclic aromatic hydrocarbons, carbon monoxide, and furans[5].

Plastic waste recycling technology encompassing both mechanical and chemical recycling offers significant benefits for resource reclamation and waste treatment. Mechanical recycling involves using mechanical processes to re-form, process, and convert waste plastics

*Author for correspondence

for reuse. However, the quality of the recycled products may be compromised due to contaminants, enhancers, and dyes in the waste plastics. Chemical recycling encompasses methods such as pyrolysis, gasification[6], depolymerization, and hydrogenation[2]. In pyrolysis, plastics are heated to 300–900 °C under oxygen-free conditions, causing polymer chains to break down into oil, gas, and coke[3,7]. This process produces minimal amounts of CO and CO_2, significantly reducing carbon emissions.

Pyrolysis of PS yields mostly aromatic compounds, with the exact composition depending on factors like pressure, temperature, reactor type, residence time, and the presence of catalysts[3,4,8]. Temperature plays a crucial role: lower temperatures tend to yield more liquid products, while higher temperatures generate more gas and coke due to increased secondary reactions[9].

Polystyrene (PS) emerges as a significant and widely utilized plastic due to its affordability and durability. Although naturally transparent, it can be infused with colorants for added versatility. With its resistance to heat, lightweight nature, and robustness, this polymer finds applications across various sectors, ranging from packaging and toys to household essentials such as computer casings and kitchen appliances. Both expanded and solid forms of PS are recyclable, yet recycling expanded polystyrene foam results in a loss of its characteristic foam texture. Although it is feasible to reintroduce gas into reclaimed polystyrene, the process escalates the product's cost compared to virgin material. Consequently, solid-state utilization prevails, particularly in molding processes.

The typical approach for treating polystyrene is pyrolysis, a process that involves thermal decomposition in the inert atmosphere to produce pyrolysis oils, gases, monomers, and other valuable chemicals[10]. Pyrolysis requires careful selection of reactors and catalysts to manage the pyrolysis temperature and determine the desired final products. Numerous studies have explored how reactor design such as batch-type, continuous flow, fixed-bed and pressure reactors affects the efficiency and outcomes of pyrolysis[11-15]. In addition, the use of solvents, various plastic blends, and different additives in the pyrolysis process has shown potential to enhance the production of liquid products[16,17].

Extensive research has focused on improving the pyrolysis of polystyrene by using acidic and basic catalysts to increase the yield and selectivity of the resulting products, while also seeking to make the process more cost-effective[18-22]. These studies aim to optimize pyrolysis techniques for better resource recovery and to reduce environmental impact by minimizing waste and reusing valuable materials.

In the present study, depolymerization of Polystyrene (PS) plastic is investigated using high alumina cement within a specialized batch reactor with a capacity of 549.77 cm^3. This reactor setup is designed to facilitate the controlled breakdown of PS into oil, gas, and other chemical components. The resulting fuel oil is subjected to Fourier-Transform Infrared (FTIR) spectroscopy and Gas Chromatography-Mass Spectrometry (GC-MS) analysis to precisely identify and quantify the chemical compounds generated through depolymerization. High alumina cement was subjected to Brunauer-Emmett-Teller (BET) analysis to understand the physical characteristics and surface area.

2.0 Materials and Method

2.1 Materials and Chemicals Used

Waste polystyrene plastic was collected from roadside dumps. The chemicals procured for catalytic pyrolysis were natural zeolite (analytical grade) and high alumina cement. A batch reactor was fabricated for catalytic pyrolysis.

2.2 Fabrication of Batch Reactor

A batch reactor of SS 304 grade material and 549.77 cm^3 capacity was fabricated for catalytic pyrolysis. The reactor was insulated with a layer of glass wool sandwiched between two layers of asbestos tape. Figure 1 represents the sectional view of the fabricated batch reactor.

2.3 Preparation of Feed

Polystyrene plastic, due to its low density, presents significant challenges for physical compression. To facilitate its handling and introduction into the batch reactor, acetone was employed as a solvent to dissolve the polystyrene, forming a slurry. The polystyrene was mixed with acetone in a 3:5 weight-to-volume ratio and the resulting slurry was exposed to sunlight for 10 minutes to allow partial evaporation of acetone. The resulting thickened slurry was used as the feed for the pyrolysis process.

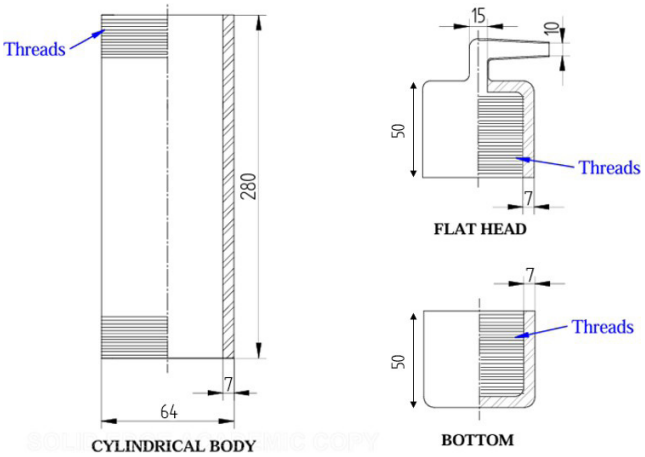


Figure 1. Sectional view of batch reactor (all dimensions are in mm).

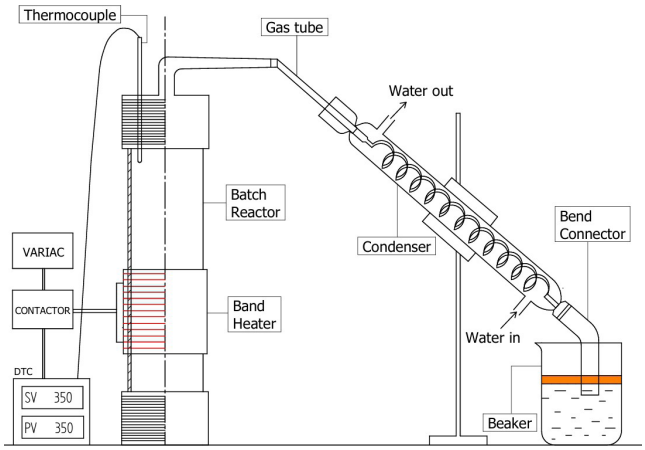


Figure 2. Schematic diagram of experimental setup.

2.4 Preparation of Catalysts

High alumina cement is a Lewis-acid type of catalyst having Si:Al ratio in the range of 0-1. It mainly constitutes of alumina, silica, calcium hydroxide, metal oxides and inorganics. It is prepared for using as a catalyst in pyrolysis process by heating at 520 °C in muffle furnace, which mainly converts the calcium hydroxide present in it to calcium oxide as indicated in Equation (1); along with disintegrating minute amounts of inorganic constituents such as $K2O_3$ SO_3.

$$Ca(OH)_2 \xrightarrow{520\ °C} CaO + H_2O \quad (1)$$

The thermal activation of catalysts is a critical step to enhance their activity, selectivity, and stability in catalytic processes. In this study, natural zeolite was activated by heating it to 250°C. High alumina cement was activated within the temperature range of 550-600°C to optimize its performance in the pyrolysis process.

2.5 Pyrolysis

Pyrolysis is a thermochemical decomposition process of organic materials at high temperatures in the absence or limited presence of oxygen. This process can be enhanced through the use of various catalysts. The catalytic pyrolysis was conducted at atmospheric pressure.

Figure 2 demonstrates the schematic of the experimental setup utilized for conducting the present study. A band heater was clamped externally to the batch reactor to serve as the heating source. Nichrome wire, wound along the inner surface of the band heater, generated the required heat through electrical resistance while voltage regulation was achieved using a variac. A K-type thermocouple, connected to a Digital Temperature Controller (DTC), was inserted through the flat head of reactor to monitor the temperature of the reaction. The DTC was set to maintain the reaction temperature at 350°C. It was connected to a contactor that automatically disconnected the power supply to the band heater upon reaching the set temperature, preventing overheating. A gas tube connected the reactor outlet to a condenser, through which cold water was continuously circulated using a pump. Initially, a bend connector at the condenser's end was dipped in water to absorb acetone. This connector was removed once oil droplets began to appear on the surface of the acetone-water mixture.

For each trial, the feed slurry was introduced from the opening of cylindrical reactor along with the thermally activated catalyst. Each catalyst was added in 10 wt.% ratio of the feed plastic. The reaction temperature was consistently maintained at 350°C. Vapours formed during the process moved through the gas tube from the reactor outlet and condensed upon contacting the condenser surface, which was maintained at 19°C. The resulting fuel oil, identified by its pale orange colour and lower density, accumulated on top of the water-acetone mixture and was separated using a separating funnel.

3.0 Results and Discussion

3.1 Products of Pyrolysis

By maintaining a constant plastic weight of 120 g and pyrolysis temperature of 350°C, notable variations in the

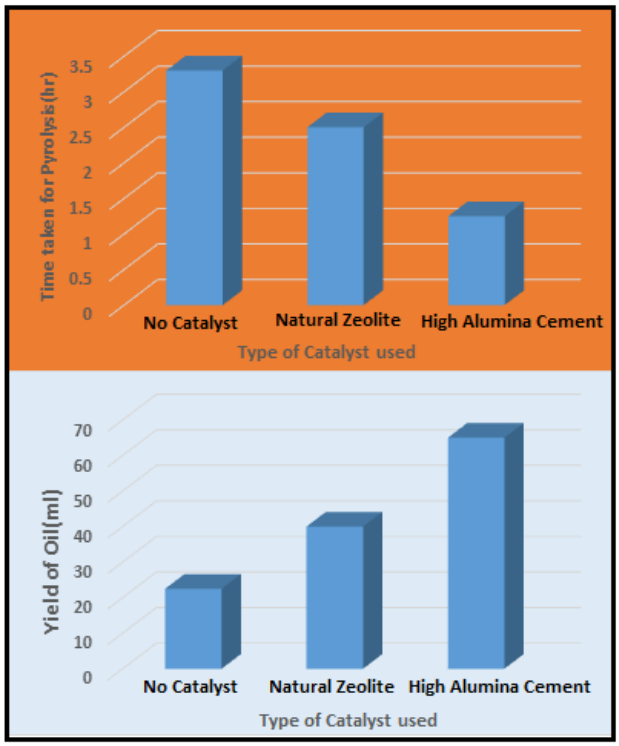


Figure3. Time taken for pyrolysis and yield of oil with and without the catalysts.

yield of fuel oil when employing different catalysts and in the absence of a catalyst was observed.

Without a catalyst, the pyrolysis process took 3.3 hours, resulting in a fuel oil volume of 22.5 ml. Introducing natural zeolite as a catalyst reduced the pyrolysis time to 2.5 hours, significantly increasing the volume of fuel oil to 40 ml. Further, employing high alumina cement as catalyst expedited the process, reducing the time to 1.25 hours, and substantially increasing the volume of fuel oil to 65 ml. The yield of fuel oil and the time taken for pyrolysis is represented in Figure 3 and summarized in Table 1 and Table 2.

3.2 Physical Properties of Fuel Oil

The physical properties of fuel oil derived using high alumina cement such as specific gravity, density, flash point and fire point were measured as reported in Table 3 .The specific gravity of fuel oil was calculated using a pycnometer. The flash and fire point of the fuel oil was measured using Pensky-Martens Closed Cup apparatus.

3.3 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to identifying compounds to determining components in a mixture. To gain insight into the composition of the oil produced, FTIR analysis was conducted, revealing the presence of various functional groups. Figure 4 represents the FTIR analysis of fuel oil obtained during thermal and catalytic pyrolysis.

The analysis unveiled the presence of aromatic components, indicating the presence of benzene rings or similar structures. Additionally, the presence of alkenes was detected, suggesting the existence of carbon-carbon double bonds within the molecular structure of the oil.

Table 1. Time taken for pyrolysis without catalyst and with catalyst

Sl. no.	Weight of plastic, W1 (g)	Catalyst used	Temperature (°C)	Time taken (hr)
1	120	No catalyst	350	3.3
2	120	Natural Zeolite	350	2.5
3	120	High Alumina Cement	350	1.25

Table 2. Yield of Fuel oil and solid residue obtained without catalyst and with catalyst

Sl. no.	Weight of plastic, W1 (g)	Catalyst used	Yield of fuel oil collected, V1 (ml)	Weight of solid residue, W2 (g)
1	120	No catalyst	22.5	57.37
2	120	Natural Zeolite	40	49.67
3	120	High Alumina Cement	65	33.67

Table 3. Physical properties of fuel oil derived using high alumina cement as catalyst

Sl. no.	Parameter	Oil derived using High Alumina Cement
1	Specific gravity	0.9068
2	Density	0.904 g/ml
3	Flash point	38°C
4	Fire point	41°C

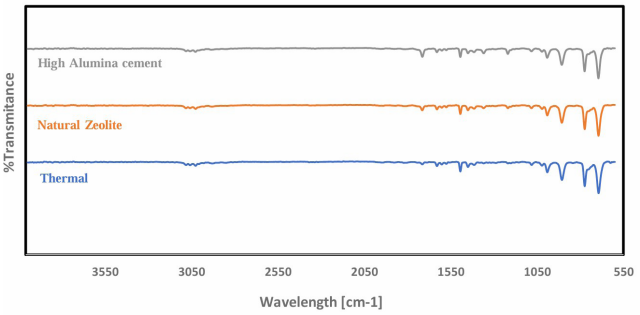


Figure 4. FTIR analysis of fuel oil obtained during thermal and catalytic pyrolysis.

The study carried out by Amjad *et. al*,[23] is referred to interpret the FTIR results. The peak observed near 2930 cm^{-1} indicates the presence of the familiar C-H bond found in most organic molecules. Near 1625 cm^{-1}, the peak signifies stretching vibrations characteristic of C=C bonds, suggesting the presence of alkenes. Within the range of 3000 to 3100 cm^{-1}, 1450 cm^{-1}, and 675 to 900 cm^{-1}, peaks indicate the existence of aromatic groups. Additionally, the around 980 cm^{-1} is attributed vibrations of CH bonds in alkene structures. Notably, the intensity of peaks within the 675–900 cm^{-1} range, indicative of aromatic groups, shows a significant increase, suggesting a rise in their concentration during the catalytic pyrolysis process.

3.4 GC-MS Analysis

Gas Chromatography-Mass Spectrometry (GC-MS) analysis is an effective analytical method utilized for identifying and quantifying chemical compounds within a sample. To obtain a detailed understanding of the specific composition of the fuel oil, GC-MS analysis was employed. This analytical technique enabled the identification of various components present in the oil along with their quantification. The majority of the components it constitutes are aromatic compounds and phenol compounds. The results revealed a diverse array of compounds, as outlined in the Table 4**.**

Table 4. Compounds present in the fuel oil obtained through GC-MS

Sl. No.	Retention time (min)	Compound name	Compound formula	Probability %
1	4.9	α-Methylstyrene	C_9H_{10}	28.7
2	5.4	(Z)-1-Phenylpropene	C_9H_{10}	24.5
3	5.6	Benzene, 3-butenyl-	$C_{10}H_{12}$	29.7
4	6	Peroxide, 1,1-dimethylethyl 1-methyl-1- phenylethyl	$C_{13}H_{20}O_2$	9.19
5	8.4	3,5-Dimethyl-4-allylpyrazole	$C_8H_{12}N_2$	24.8
6	9.3	Benzoic acid, 2-phenylethyl ester	$C_{15}H_{14}O_2$	41.3
7	11	Diphenylmethane	$C_{13}H_{12}$	13.9
8	12.2	Bibenzyl	$C_{14}H_{14}$	42.7
9	14	(+)-N-Benzyl-α-phenethylamine	$C_{15}H_{17}N$	20.8
10	15	Naphthalene, 1,2,3,4-tetrahydro-2-phenyl-	$C_{16}H_{16}$	27.9
11	16.1	cis-4-Benzyl-2,6-diphenyltetrahydropyran	$C_{24}H_{24}O$	11.4
12	16.4	2H-1-Benzopyran-4-ol, 3,4-dihydro-2-(4- methoxyphenyl)-	$C_{16}H_{16}O_3$	5.4
13	16.6	2-Naphthalenamine, 1,2,3,4-tetrahydro-N- (1-methylethyl)-N-(phenylmethyl)-	$C_{20}H_{25}N$	23.5
14	17	Naphthalene, 1-(phenylmethyl)-	$C_{17}H_{14}$	18
15	17.9	Naphthalene, 1,2,3,4-tetrahydro-1-phenyl-	$C_{16}H_{16}$	33.4
16	20.2	Cyclohexane, 1,3,5-triphenyl-	$C_{24}H_{24}$	20
17	20.5	N,N'-Bis (salicylidene) ethylenediiminocobalt (II)	$C_{16}H_{14}CoN_2O_2$	1.95
18	21.3	2-Cyclohexen-1-one, 4,4-diphenyl-	$C_{18}H_{16}O$	16.9
19	21.6	2-Quinolinol, 6-bromo-3-cyano-4-phenyl-	$C_{16}H_9BrN_2O$	7.14
20	21.7	Dicentrine	$C_{20}H_{21}NO_4$	4.47
21	22.2	Benzene, 1,1'- (1-heptenylidene) bis-	$C_{19}H_{22}$	10.2
22	24.2	Acetamide, 2-phenyl-N-benzyl-N-nonyl-	$C_{24}H_{33}NO$	4.37
23	24.7	1-Naphthalenemethanol, heptafluorobutyrate	$C_{15}H_9F_7O_2$	7.28

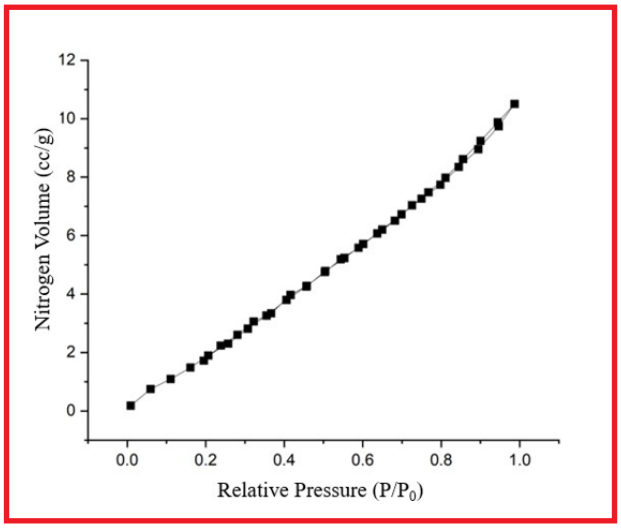


Figure 5. Nitrogen adsorption-desorption plot of high alumina cement.

3.5 BET Analysis

BET analysis is a method employed to assess the surface area of materials, notably porous solids such as catalysts and adsorbents. Figure 5 demonstrates the BET analysis of high alumina cement catalyst.

To comprehensively assess the surface properties of high alumina cement as catalyst, it underwent BET analysis, revealing a surface area of 17.802 m^2/g. This notably exceeds the surface area of natural zeolites, which is 13.69 m^2/g [24], and the other parameters were compared as shown in Table 5. We can observe that the pore volume of high alumina cement is lower than that of the natural zeolite. The lower the pore volume, the higher the surface area and this is confirmed from the values of pore volume indicated in Table 5.

The significantly larger surface area of high alumina cement catalyst suggests a greater availability of active sites for catalytic reactions, thereby enhancing its performance in the conversion of waste plastic to fuel oil. This finding aligns with the observed superior efficiency of high alumina catalyst compared to natural zeolite in the catalytic pyrolysis process. The increased surface area not only contributes to improved catalytic activity but also holds promise for further optimization and fine-tuning of the catalyst's properties for enhanced performance in plastic waste conversion.

Table 5. Surface area, pore volume and pore diameter of catalysts by BET

Parameter	Natural Zeolite[24]	High Alumina Cement
BET surface area, m^2/g	13.69	17.802
Total pore volume, cc/g	0.032	0.020
Average pore diameter, nm	17.45	2.856

4.0 Conclusion

A comparative analysis of thermal and catalytic pyrolysis processes was conducted using a fabricated batch reactor, revealing the high efficiency and superiority of the high alumina cement used as a catalyst over conventional natural zeolite. Initially, thermal pyrolysis within the stainless-steel reactor produced 22.5 ml of oil in 3.3 hours. Catalytic pyrolysis with natural zeolite improved the production to 40 ml in 2.5 hours. Remarkably, using the high alumina cement as catalyst resulted in a substantial increase in oil production to 65 ml in just 1.25 hours. This significant enhancement in both yield and process efficiency clearly demonstrates the superior performance of high alumina cement in converting waste plastic feedstock into valuable oil.

The composition of the fuel oil was characterized using GC-MS and FTIR techniques. Additionally, the cost-effectiveness of high alumina cement positions it as a viable alternative to natural zeolite. This study highlights the potential of high alumina cement used as catalyst in promoting solutions for plastic waste management, offering both superior performance and economic feasibility, and advancing resource recovery and utilization. Future investigations may explore the deployment of a broad spectrum of cost-effective catalysts, along with the integration of semi-batch or continuous flow reactor configurations, to enhance the efficiency and scalability of waste polystyrene pyrolysis.

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6.0 Contribution of Authors

Conceptualization, H A Deepa; methodology, Chandini N V, Himanshu Mishra, Priyanka Sahu, R Neelesh; validation, H A Deepa.; formal analysis, H A Deepa.;

investigation, Chandini N V, Himanshu Mishra, Priyanka Sahu, R Neelesh; resources, R Ravishankar ; data curation, R Ravishankar; writing—original draft preparation, Chandini N V, Himanshu Mishra, Priyanka Sahu, R Neelesh; writing—review and editing, H A Deepa, Vikas G N; visualization, H A Deepa; supervision, R Ravishankar. All authors have read and agreed to the published version of the manuscript.

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