Synthesis of Catalyst for Effective Degradation of Waste Plastics

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Abstract— Municipal plastic wastes from an environmental hazard due to its non-biodegradable nature. Municipal plastic wastes mainly composing of polypropylene as the polymer were pyrolysed at a temperature of 400-450oC. Liquefaction followed fast pyrolysis in a batch reactor under an inert (nitrogen) atmosphere. A composite metal oxide such as NiO, Al2O3, MgO, Ni-Al((1:1) Ratio, mole%), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide was synthesized for the process. Comparative studies for the amount of oil obtained in absence and presence of catalyst were carried out. The prepared composite metal oxide was tested for surface properties by X-Ray Diffraction and Scanning Electron Microscopy. The yield was improved while using composite metal oxide catalyst. Effect of catalyst on temperature and residue was studied. This method proves to be an environmental friendly approach to degrade plastic wastes and proves to be an alternative source of fuel.

Key words: Polypropylene, Pyrolysis, Composite Metal Oxide, Degradation

I. INTRODUCTION

Plastic products are necessary in our life. With increasing number of applications being found for plastic materials, the majority uses is in packaging and house hold products. Chennai region generates 4248 tons/day of solid waste; from that 66% wastes are non-biodegradable. In current scenario more than 3% wastes are plastics. The plastic waste contains 42.3% of polypropylene, 42% of polyethylene, 7% polystyrene, 8.7% of polyester and others [Rajendra Kumar Kaushal et al., 2012]. Polypropylene waste is more in Chennai region, so only Polypropylene is selected for the study.

Pyrolysis is a thermal process in the absence of oxygen. The polymeric materials are heated to high temperatures, so their macromolecular structures are broken down into smaller molecules and a wide range of hydrocarbons are formed. These pyrolytic products can be divided into a gas fraction, a liquid fractions consisting of paraffin's, olefins, naphthenic and aromatics, and solid residue [Baoyingshi et al., 2012].

The Polypropylene pyrolysed with Fe/AC (5%Fe/AC), Non catalyst +CS_2 (0.3 wt. %), Fe/AC+CS_2 (5%Fe/AC+ 0.3wt%) catalyst [Ikuseina Kamura et al., 1996], ZSM-5 and red mud (Fe_2O_3-36.5%, Al_2O_3-23.8%, TiO_2-13.5%, SiO_2-8.5%, CaO-5.3%, Na_2O-1.8%, Others-10.6%) as a catalyst [Lopez A et al., 2011], Cao, $Zncl_2$, Fe, PZSM-5(Si/Al: 25, Si/Al: 38, Si/Al: 50) catalyst in the ratio of 0.8:10wt%. [Baoyingshi et al., 2012], Alumina loaded mesoporous catalysts (Al-MCM-41 and ALS-SBA-15 [Zeynep Obali et al., 2012], Ni used as a catalyst [Neeraj Mishra et al., 2013], Ni-Mo, $Al(OH)_2$, HZSM-5, Zeolite are used as a catalyst [Norbert Miskolczi et al., 2013], Pt/Al_2O_3 used as a catalyst [Ewa Smigiera et al., 2014] to get more amount of liquid product and to reduce residue of the raw material and to reduce the process temperature.

Usage of commercial and synthetic catalysts reduces the formation of residue of the polymer. By using catalyst the degradation temperature also gets reduced. In earlier literatures mostly metal catalyst were used. A metal catalyst after calcinations (NiO-Al_2O_3) gave better result. Compared to single compound metal oxide, combination of metal oxides or composite metal oxides gave better results; hence composite metal oxide catalyst was selected for the current work.

Recent research tells us while doing pyrolysis process plastics/polymers are converted into light hydrocarbon but only the defects is maximum yield can't obtained. Till now pyrolysis process done only with pure polymers such as polypropylene, polyethylene, polystyrene, and poly vinyl chloride crystals obtained from plastic industries. But in this study based on waste plastic's obtained from home waste.

II. EXPERIMENTAL

A. Materials

Plastic chair (Polypropylene) is the source for study and it was collected from dustman. To prepare a catalyst sulphate salts are used. Source of Ni is $NiSO_4.6H_2O$ with the Assay of min 98% and it was purchased from LOBA Chemie Laboratory Reagents & Fine chemicals, Mumbai. Source of Al is $Al(SO_4)_3.18H_2O$ with the purity of 51-59% and which is purchased from LOBA Chemie Laboratory Reagents & Fine chemicals, Mumbai. Source for Mg is $MgSO_4.xH_2O$ with the purity of min 99% (dry basis) and it was purchased from SISCO Research Laboratories Pvt. Ltd, Mumbai. Solvent used for the process is NH_4 (OH) analytical reagent with the Assay of min 25% which is from SISCO Research Laboratories Pvt. Ltd, Mumbai. Distilled water with pH of 6.95 and which is from Modern Disleries and chemicals Pvt. Ltd, Chennai. Ammonia buffer solution with the pH of 10.5 and it was purchased from RFCl Limited, New Delhi.

B. Preparation of Catalyst

Preparation of catalyst includes two methods. They are impregnation and precipitation method. The precipitation method is the best method for composite metal oxide catalyst preparation compared to impregnation method; because precipitation method is based on capillary forces, whereas the precipitation method is based on pH maintaining technique. Ni-Al-Mg ((1:1:1) Ratio, mole%) catalyst was prepared by pH maintaining technique in which 1M of $NiSO_4.6H_2O$, $Al(SO_4)_3.18H_2O$ and $MgSO_4.xH_2O$ was taken and equal volume of 1M of $NH_4(OH)$ was added and stirred for 12h with the pH (final pH value of the solution) of 12, 9, 10, 7.9, 8.2, 9.5 and 8.3 (±0.1for all) for NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1) and Ni-Al-Mg(1:1:1) respectively. The precipitation was carried out (at 40°C for NiO, MgO, Ni-Al(1:1), Ni-Mg(1:1), and Ni-Al-Mg(1:1:1) at 100°C for Al_2O_3 and Al-Mg(1:1)) and cooled to room temperature then washed with water and drying at 105°C for

10h. Finally the catalyst was calcined at 750ºC for 3h [Toshio MAKI, 1981; Tomohiro Nakayama et al., 1997; Sumit Banerjee et al., 2003; Chunfei Wu et al., 2009; YI Jian-hong et al., 2009; Li-Zhai pei et al., 2010].

C. Process description

Pyrolysis is done with the one liter capacity of pyrolysis reactor. The Source of waste plastic chair (polypropylene) is taken for the process. The plastic chair is shredded into average size of 2-4cm. 100g of sample was loaded to the reactor per batch and the catalyst weight vary from 0.5g to 3g. The reactor temperature adjusted up to 450ºC. The vapor from the reactor is condensed by double pipe condenser by supplying cold water. The cooling water temperature is 10ºC and the liquid product was collected at bottom. The temperature at which the first drop of oil is collected was maintained for 30 minutes. Non condensable gas was collected at the top of the liquid collector tank.

III. RESULTS & DISCUSSION

The volatile nature of plastic sample is analyzed through thermo gravimetric analysis (TGA). Lattice structure and morphology of the catalyst can be noticed through XRD and SEM.

A. Raw Material Analysis

Fig.1 shows the thermo gravimetric analysis of plastic chair, which indicates the volatile nature of plastic. The volatile products of plastic were obtained from 163.66ºC to 476.21ºC and the maximum weight loss occurred at 452.57ºC. At this temperature, 76.89% of sample losses its properties (i.e. evaporated) and up to this temperature 83.187% of sample gets evaporated. The weight loss was not significant above 650ºC and yielded a residue of 6.722%. Derivative weight percentage of plastic was a maximum at 452.5ºC which indicates the temperature above which the plastic would be converted to volatile matter.

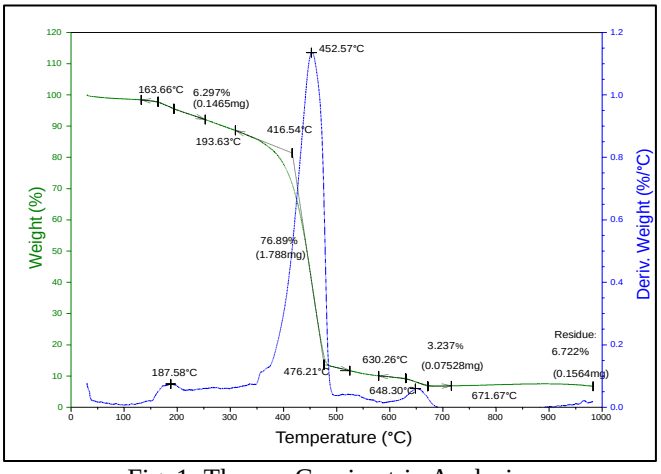


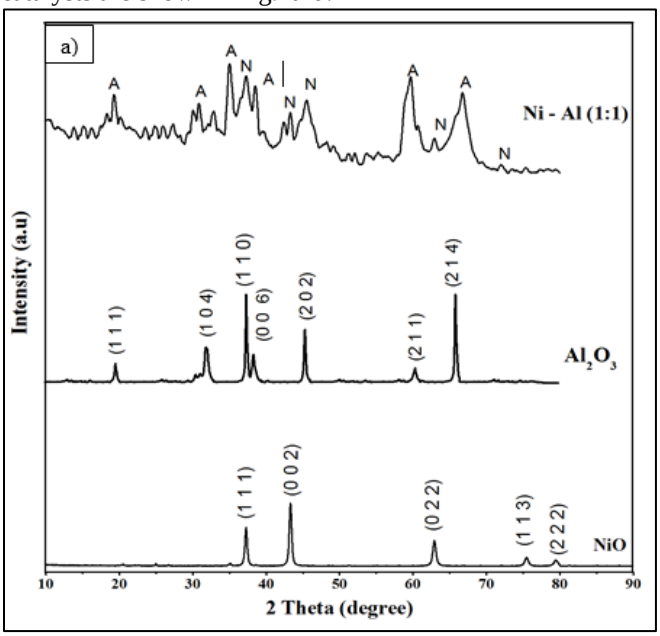
Fig. 1: Thermo Gravimetric Analysis

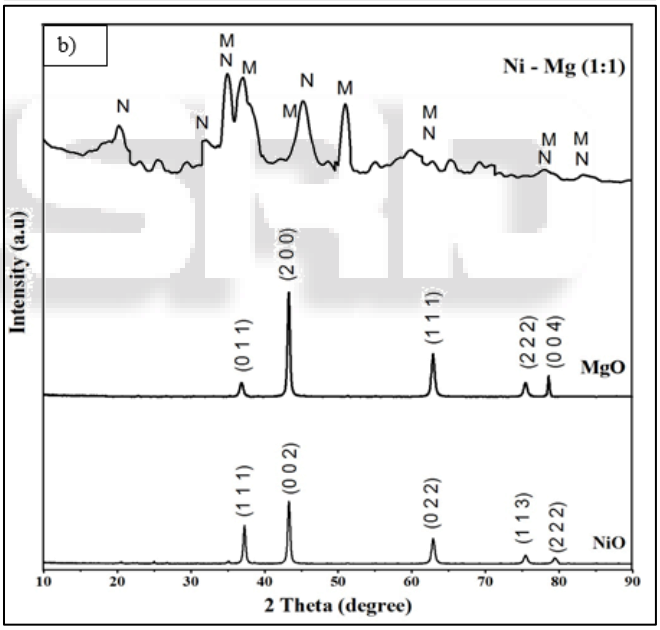
B. Characterization of catalyst

The prepared catalyst NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide were analyzed by SEM (Scanning Electron Microscopy) and XRD (X-Ray Diffraction) analysis. SEM image shows the shape and morphology of catalyst. XRD graph shows the components and its crystalline phase nature.

1) X-Ray Diffraction Analysis

X-Ray Diffraction analysis of NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalysts are shown in fig.2a-d.





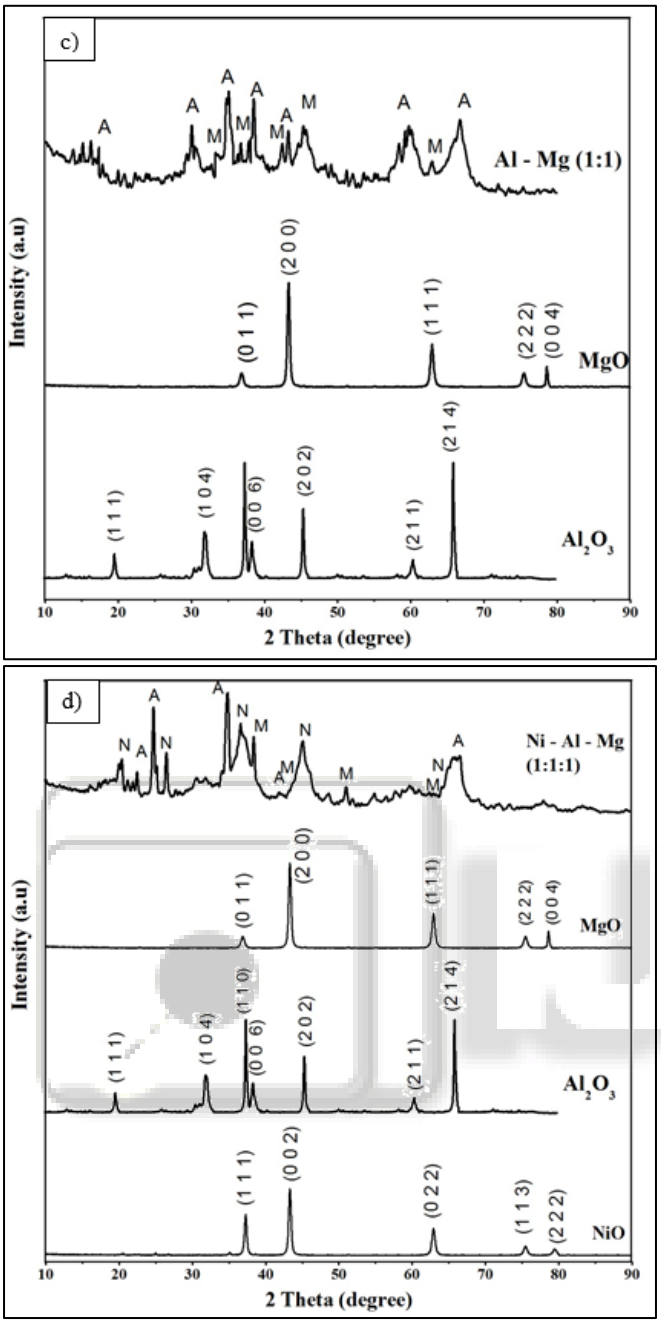


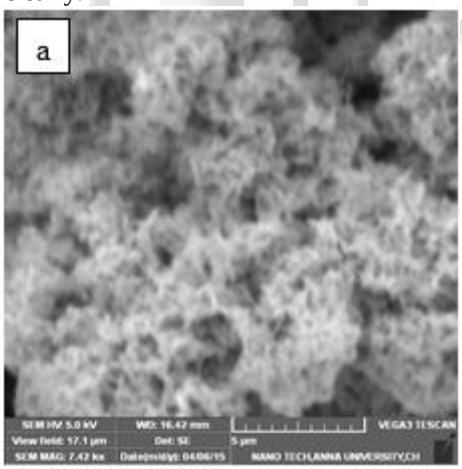
Fig. 2: X-ray Diffraction Analysis of a) Ni-Al (1:1) oxide b) Ni-Mg (1:1) oxide c) Al-Mg (1:1) oxide d) Ni-Al-Mg (1:1:1) oxide

NiO, Al_2O_3 and MgO catalysts gives clear peaks at the corresponding diffraction angle and these peaks directly matches with JCPDS card number 901-2319, 29-0063 and 45-0946 respectively. NiO, Al_2O_3 and MgO catalysts shows the homogeneous phase structure. Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalyst results the heterogeneous phase structure. Corresponding JCPDS card number for Ni-Al (1:1) and Al-Mg (1:1) oxide was 900-1433 and 21-1152 respectively. Ni-Mg (1:1) oxide catalyst matches with JCPDS card number 14-0117, 100-054. Ni-Al-Mg (1:1:1) oxide catalyst matches with JCPDS card number 101-0382, 100-0033, 100-054, 500-0226, 900-2349 and 901-2319. Ni-Al (1:1) oxide catalyst is found to contain oxide such as NiO, Al_2O_3, and as well as Ni-Al_2O_3. XRD result of Ni-Mg (1:1) oxide catalyst shows the presence of oxides such as NiO, MgO and Ni-MgO. Al-Mg (1:1) oxide catalyst shows the presence of oxide such as Al_2O_3, MgO and Al-MgO. Ni-Al-Mg (1:1:1) oxide catalyst shows the presence of oxide such as NiO, Al_2O_3, MgO, Ni-Al_2O_3, Al-MgO and Ni-MgO. The sharp peaks represents crystalline phase of metal oxides and the small peaks represents impurities.

The average particle size dispersion for NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalyst was found to be 29.8nm, 38.9nm, 34.6nm, 48.9nm, 41.3nm, 39.4nm and 34.9nm respectively. The catalyst sizes obtained were in the range of mesoporous (2nm to 50nm).

2) SEM Analysis

SEM analysis of NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalysts shown in Fig.3a-g. SEM analysis is done with low magnification and view field in the range of microns. By making the composite the conductivity of catalyst gets changed so; the composite catalyst SEM images are taken with low magnification. At high magnification the SEM images are not clear. SEM image shows the different size of crystals with morphology of catalyst. NiO, Al_2O and MgO catalyst shows a homogeneous phase structure with less than micron sizes crystals. Fig.6d-g shows the Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalysts were heterogeneous in structure and the crystal sizes ranging from nanometer size particles to quite big ones formed by agglomeration of the crystals of catalyst, which confers a highly heterogeneous internal structure of catalyst and non-linear arrangement of crystals. Fig.3a-g SEM images were obtained in micron size of view field, from XRD analysis all the catalyst sizes in mesoporous range (2nm to 50nm) so only the catalyst structure was not obtained clearly.



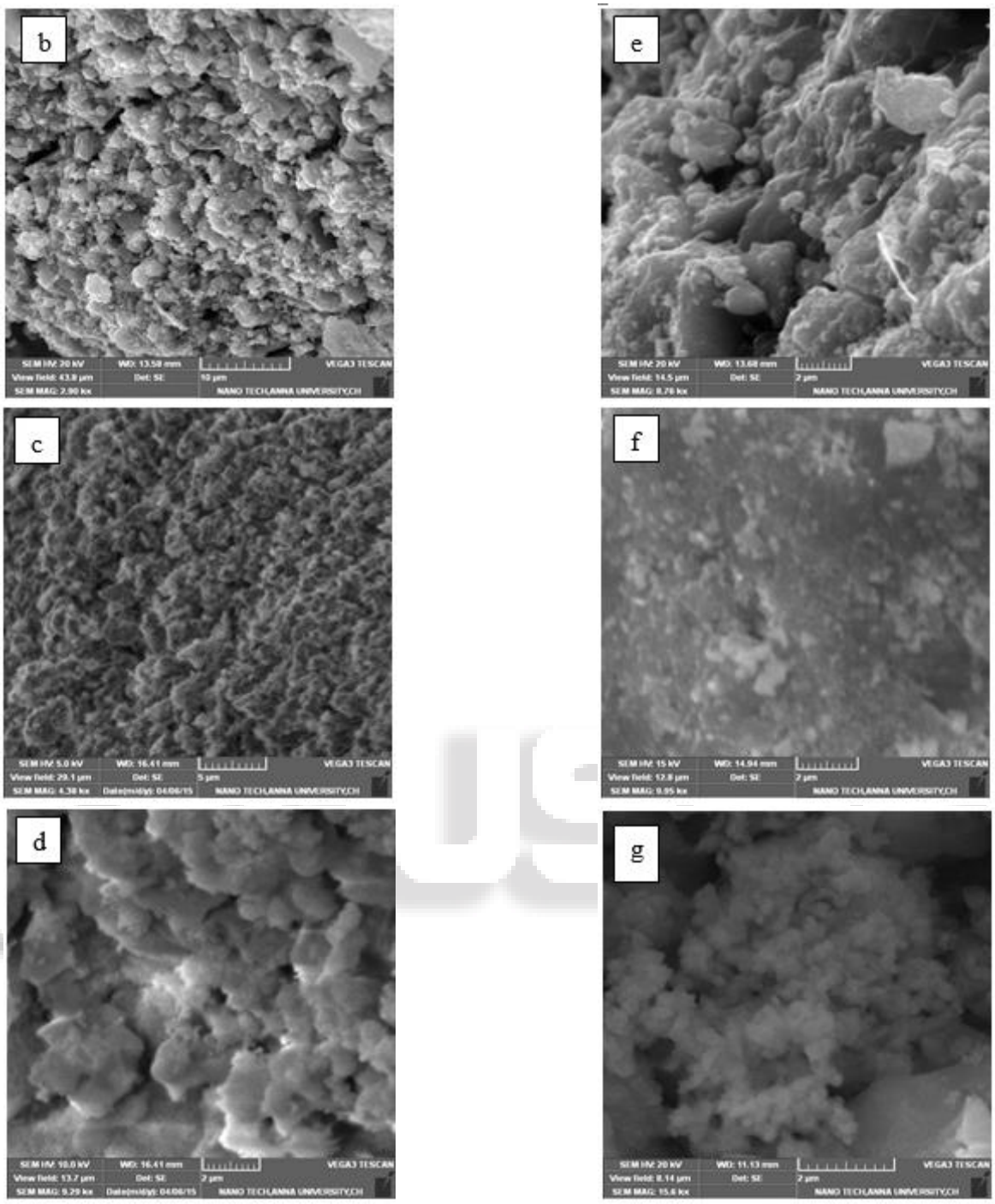


Fig. 3: SEM Analysis of a) NiO catalyst b) Al2O3 catalyst c) MgO catalyst d) Ni-Al(1:1) oxide catalyst e) Ni-Mg(1:1) oxide catalyst f) Al-Mg(1:1) oxide catalyst g) Ni-Al-Mg(1:1:1) oxide catalyst

C. Yield pyrolysis products

The pyrolysis of plastic has been carried out with and without catalyst and the yield of the products obtained and the temperature at which this pyrolysis was carried out is shown in Table 1.

S.no	Process	Yield %			Process Temperature (°C)	Process Time (min)
		Oil	Gas	Residue		
1	Thermal	66.20	18.8	15.00	450	30
2	NiO	67.03	18.41	14.56	410	30
3	Al_2O_3	69.28	17.71	13.01	410	30

4	MgO	70.17	16.33	12.65	400	30
5	Ni-Al oxide (1:1)	73.27	13.63	13.10	400	30
6	Ni-Mg oxide (1:1)	68.87	17.49	13.64	410	30
7	Al-Mg oxide (1:1)	69.35	17.19	13.46	410	30
8	Ni-Al-Mg oxide (1:1:1)	77.57	10.80	11.63	390	30

Table -1 Product in % of mass

The products were essentially oil, gas, and residue however, the emphasis of the current works in to maximize the yield of the liquid product at the lowest processing temperature. The tabulated values are obtained at 1g, 2g, 1g, 1g, 1g, 0.5g and 3g of catalyst weight for NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalysts respectively because, the process temperature does not changed after these weight (1g, 2g, 1g, 1g, 1g, 0.5g and 3g) of catalyst when used for the pyrolysis process. In the absence of catalyst yield% of oil, residue, non-condensable gases are 66.20, 15.00, 18.80 respectively and the process temperature were 450°C. By using different catalysts the yields of desired and undesired product yields are varied. NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalyst was used for the pyrolysis process.

By using NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide as a catalyst yield of desired product gets increased to 67.03%, 69.28%, 70.17%, 73.27%, 68.87%, 69.35% and 77.57% respectively at the same time process temperature was reduced to 410°C, 410°C, 400°C, 400°C, 410°C, 410°C and 390°C respectively. The residue of plastic obtained in the absence of catalyst was 15.00%. In the presence of NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalysts, residue of plastic gets reduced to 14.56%, 13.01%, 12.65%, 13.10%, 13.64%, 12.46% and 11.63% respectively. Yield percentage of non-condensable gases are also slightly reduced with all the catalyst here what we used. The pyrolysis process was carried out for 30min.

1) Effect of Catalyst on Temperature

Fig.4 shows the effect of catalyst on the pyrolysis temperature. By using NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1) and Ni-Al-Mg (1:1:1) oxide catalysts the process temperature gets reduced. The catalyst reduces the process temperature with different ratio of catalyst because, of their different active nature (activity). By using NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalysts process temperature was reduced to 410°C, 410°C, 400°C, 400°C, 410°C, 410°C and 390°C respectively. From the graph best result was found to be 390°C with Ni-Al-Mg (1:1:1) oxide catalyst when 3g of catalyst used.

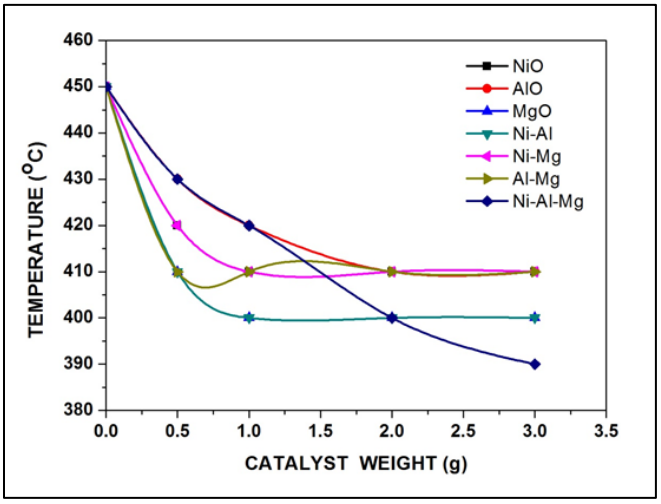


Fig.4: Effect of catalyst on temperature

The same result was obtained while increasing the Ni-Al-Mg (1:1:1) oxide catalyst weight more than 3g. The process temperature was the least when the catalyst loading was 1g, 2g, 1g, 1g, 1g, 0.5g and 3g of catalyst for NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide respectively.

2) Effect of Catalyst on Residue

Fig.5 shows the effect of catalyst on the amount of Residue with different ratio of catalyst. For NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalysts residue of plastic after pyrolysis process gets changed.

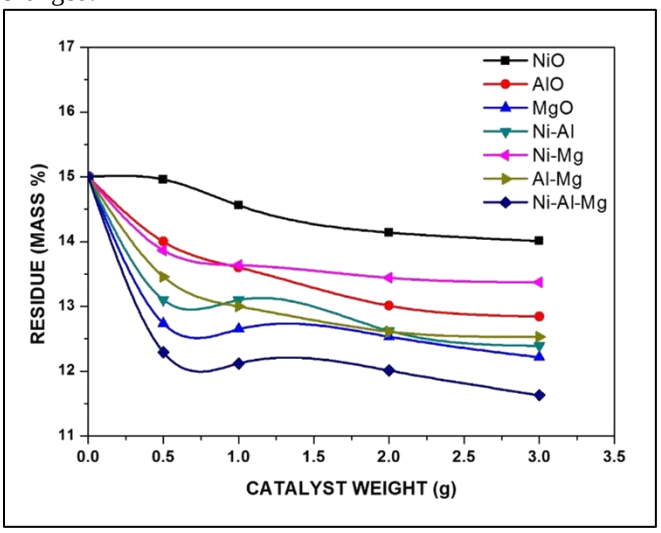


Fig.5 Effect of catalyst on residue

The NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalysts showed least residue when the catalyst was 1g, 2g, 1g, 1g, 1g, 0.5g and 3g respectively. In the presence of NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalyst, residue of plastic gets reduced to 14.56%, 13.01%, 12.65%, 13.10%, 13.64%, 12.46% and 11.63%

respectively. From this graph the best catalyst was found to Ni-Al-Mg (1:1:1) oxide, because it reduces the residue of plastic to 11.63% at 3g of catalyst.

3) Effect of Catalyst on oil yield

Fig.6 shows the effect of catalyst on the oil yield with different ratio of catalyst. For NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1:1) oxide catalysts yield of plastic oil is optimized when the catalyst ratio was 1g, 2g, 1g, 1g, 1g, 0.5g and 3g respectively. In the presence of NiO, Al_2O_3, MgO, Ni-Al (1:1), Ni-Mg (1:1), Al-Mg (1:1), and Ni-Al-Mg (1:1) oxide catalyst, oil yield was found to 67.03%, 69.28%, 70.17%, 73.27%, 68.87%, 69.35% and 77.57% respectively. The maximum percentage of oil yield obtained when Ni-Al-Mg (1:1) oxide catalyst used.

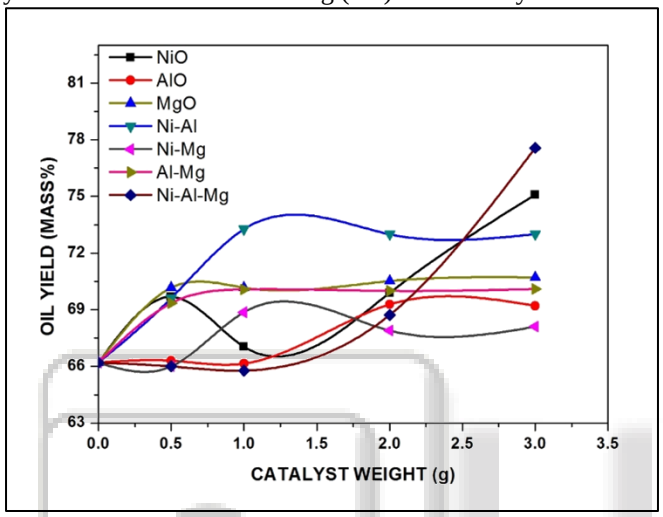


Fig. 6: Effect of catalyst on oil yield

IV. Conclusion

The synthesized NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalysts has different activity. Because, of it the process temperature gets reduced. All the catalyst here we used reduces the process temperature. The efficient result was found for Ni-Al-Mg(1:1:1) oxide catalyst and it reduces the process temperature upto 390°C.

The desired product of pyrolysis process was mostly liquid (plastic oil) and non-condensable gases also has the valuable usage. Undesired product is residue of plastic. The yield of the liquid product was found to increase by using the NiO, Al_2O_3, MgO, Ni-Al(1:1), Ni-Mg(1:1), Al-Mg(1:1), and Ni-Al-Mg(1:1:1) oxide catalysts. The effective result was found from Ni-Al-Mg(1:1:1) oxide catalyst and it gives the 77.57% of liquid (plastic oil) product, 10.80% of gaseous product and 11.63% of residue.

From the synthesized metal oxide and composite metal oxide catalysts, best results found from Ni-Al-Mg (1:1:1) oxide catalyst, because it gives more amount of liquid product at lower processing temperature.

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