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Carbon Nanotubes Based Porous Framework for Filtration Applications Using Industrial Grinding Waste

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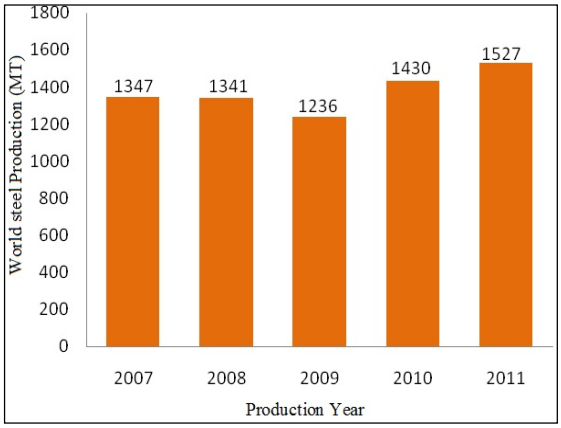
***Abstract*—**Forging, milling, turning, grinding and shaping etc. are the various industrial manufacturing processes which generate the metal waste. Grinding is extensively used in the finishing operation. The waste generated contains significant impurities apart from the metal particles. Due to these significant impurities, it becomes difficult to process and gets usually dumped in the landfills which create environmental problems. Therefore, it becomes essential to reuse metal waste to create value added products. Powder injection molding process is used for producing the porous metal matrix framework. This paper discusses the presented design of the porous framework to be used for the liquid filter application. Different parameters are optimized to obtain the better strength framework with variable porosity. Carbon nanotubes are used as reinforcing materials to enhance the strength of the metal matrix framework.

***Keywords*—**Grinding waste, powder injection molding, carbon nanotubes, metal matrix composites.

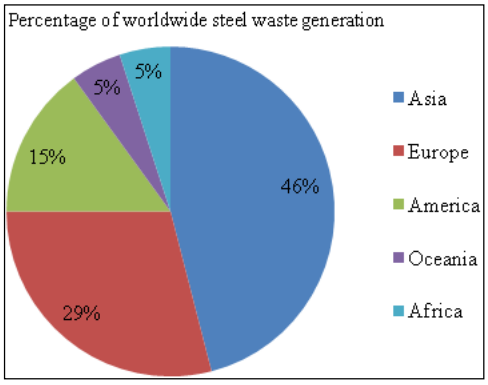
I. Introduction

DEVELOPMENT of the steel industry has brought environmental degradation due to rapid and extensive industrialization and urbanization. The China consumes much more steel as compared to India; however, the Indian steel consumption may rise to 110 kg to 300 kg from 2020 to 2030. These projections indicate that steel requirement will increase in the near future. This leads to increase the waste generation which creates environmental issues if not properly treated [1].

The current steel waste generation is around 5.67 million tones worldwide [1]. In India, iron waste generated is 0.33 million tones (5.48% of world waste) [1]. Figs. 1 (a) and (b) show the world steel production and steel waste generated in different countries respectively. Metal waste treatment poses a great threat to the environment and required significant energy to make this waste reusable. The different processes are used to create the value added products from metal waste, such as Powder Injection, Melting and Solidification, and Electrochemical Deposition. Powder Injection route is most favorable as it requires less tooling as well as least energy consumption as compared to all another process. In powder injection molding (PIM), carbon nanotubes (CNTs) are used as a reinforcing materials as it possesses excellent mechanical properties. The tensile strength of iron is about 22 to 25 MPa whereas that of CNT is 50 to 60 GPa [2]-[5].



(a)



(b)

Fig. 1 (a) World steel Production in MT (2007-2011) and (b) Steel waste generated worldwide [1]

Powder injection process offer advantage to create the product close to the accurate dimensions thus reduces the waste generation. The unique features of the process make it an attractive route for the fabrication of metal matrix composite materials [2]. Powder injection molding begins with very fine powders blended with polymers and produced pelletized feedstock. The binder holds particles in the green sample which is necessary to provide the strength before debinding. The debinding process is used to remove the

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binder, followed by sintering to form a fully dense component [3]-[7].

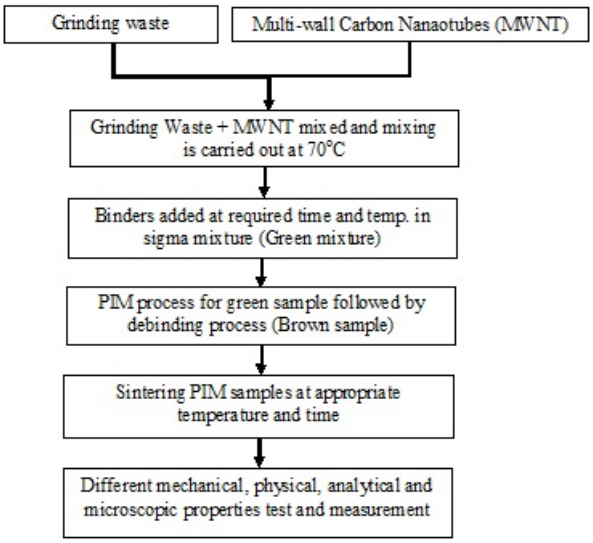

Fig. 2 Metal Matrix Composites fabrication process

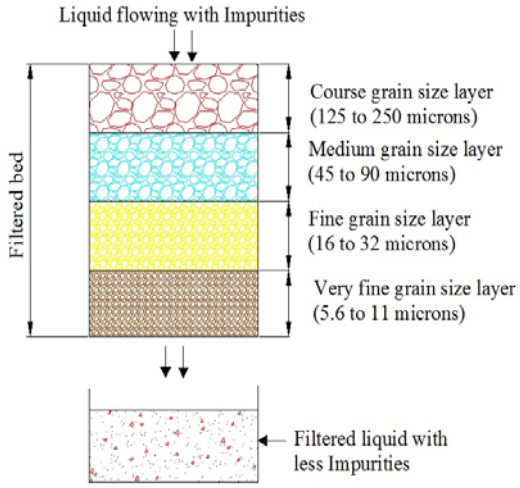

Fig. 3 Different grain size layers for liquid filtration framework

Solvent debinding is a binder removing process which keeps the component rigid without chemical reactions. Lower temperatures minimize defects and distortions [8]. Different studies [6]-[8] are dealt with improvement in the properties of the metal matrix composite formation.

CNTs withstand the extrusion force but they found non-uniform dispersion and inhomogeneity in the composite matrix [9]. The higher percentage of the CNTs in the mix creates agglomeration problem causes inferior mechanical properties. They suggest that 1 wt% CNTs gives better quality composite as compared to the 2 wt% CNTs mix [10]-[12]. Bakshi et al. review critically about the carbon nanotube-based metal matrix and suggested that the powder metallurgy route is the most popular method for CNTs based metal matrix composite preparation as compared to other methods available [13]. This method is quite simple and involves lower temperature sintering which avoids damage of CNTs and gives better quality products. Metal matrix composites are commonly used in automobile applications as connecting rod, filter, piston pin, and cylinder liner [14].

Aqidai et al. concluded that porosity formation is largely caused by mixing speed of mixture, gas entrapment during vigorous stirring and shrinkage during solidification of MMC [15]. The presence of porosity, consequently, decreases the mechanical properties of MMCs. With the addition of CNTs into MMCs, manufacturers can manipulate a material's strength, flowability, porosity, thermal stability and weight of the product [16]. Porosity plays a significant role in dynamic flow properties of the liquid. The different grain size layers separate micro size impurities from the liquid which has an important effect on service life in practical application [17].

This paper contributes towards the utilization of metallic grinding waste for the production of porous MMCs which will be helpful for the fabrication of filtration system.

II. Methodology and Experimentation

In order to design the porous framework, following objectives have been formulated.

A. The effect of different binders and change in the percentage of binders to get good flowability during injection as well to ensure the good dispersion of CNTs in the green mixture.
B. The effect of time and temperature to examine the strength of the green sample.
C. Optimize sintering time and temperature for PIM products.
D. Testing of physical/mechanical properties of PIM product.
E. Measurement of microscopic/analytical properties of the MMCs to produce good strength PIM product as per the application intended for.

For the production of MMCs, different parameters such as loading of grinding waste, the percentage of carbon nanotubes, time required for sigma mixing, kind of debinding, sintering temperature and time, etc. can be optimized. The impurities present in the grinding waste act as a backbone to the green mixture. An addition of stearic acid transfers some of the impurities in the waste into a cellulose kind of material. However, the mechanical strength of these PIM products will be inferior. The addition of CNTs in appropriate proportion (1% to 6% by weight) improves the properties. Table I shows the raw materials used for a preparation of Grinding Waste-CNT composite with mixing percentage and their weights percentages. Fig. 2 explains the process for the fabrication of MMCs and Fig. 3 shows the different grain size layers as per ASTM, to filter the contaminated liquid with different size impurities present in the liquid. Generally, filter pore size is around 30 micron. 99% particle removal that are 10 micron in size, 98% particle removal that are 7 micron in size, 95% particle removal that are 5 micron in size. Filter will remove about 72% of particles in 8 to 10 micron range, about 50% of particles in 20 to 40 micron range but only about 24% in 8 to 10 micron range. Table II shows ASTM grain size numbers of the metal particles corresponding to average diameters in microns required for the filtration applications.

TABLE I
MIXING PROPORTION OF RAW MATERIALS IN WEIGHT %

Material (Wt.%)	Sample A (85-15)	Sample B (87-13)	Sample C (89-11)	Sample D (90-10)	Total Requirement
Grinding Waste	146.16 gms	146.16 gms	146.16 gms	146.16 gms	584.64 gms
CNT	1.74 gms (1%)	5.22 gms (3%)	8.70 gms (5%)	10.44 gms (6%)	26.10 gms
BINDERS	26.10 gms	22.62 gms	19.14 gms	17.40 gms	85.26 gms
Total	174 gms	174 gms	174 gms	174 gms	696 gms

TABLE II
ASTM GRAIN SIZE NUMBERS

ASTM No.	1	2	3	4	5	6	7	8	9	10	11	12
Average Diameters (Microns)	250	180	125	90	65	45	32	22	16	11	8	5.6
Relative Sizes		Coarse			Medium			Fine			Very fine	

Fig. 4 (A) Fabricated sigma mixer with Green Mixture, (B) Schematic diagram of furnace used for sintering, (C) Green filter sample with dies and (D) Brown filter sample

Fig. 4 (a) shows sigma mixer used for homogeneous mixing of raw materials along with heating at appropriate temperature to impart the flowability. It is necessary to control the speed of mixing blades to reduce the friction between wall of sigma mixture and metal particles during mixing. It shows green mixture discharged by tilting bowls. This green mixture is injected by maintaining 100 psi pressure and 650 °C temperature to prepare green samples which are used to temperature to prepare green samples which are used for various mechanical/microscopic/analytical properties measurement. After injection molding, binder is removed from the green samples by solvent debinding process. Improper debinding leads to distortion and produces defect inside the sample. Solvent debinding process is selected to remove the impurities. The liquid debinding carried out in the n-heptane solution under the sonication bath for 2 hours at 60 °C bath

temperature. After completion of process, samples are removed and allowed to dry. These samples are termed as brown samples. The brown samples are weighed before and after debinding and compared with the expected target mass to verify the degree of debinding occurred. Fig. 4 (b) shows electric chemical vapor deposition (CVD) furnace used for sintering. All experiments are carried out in an argon atmosphere at a constant flow rate of 100 ml/min. Inert atmosphere prevents the oxidation of brown samples. During sintering, solid-state atomic diffusion takes place, followed by recrystallization and grain growth. The samples obtained from the debinding process are placed in the quartz tube of the furnace and heated gradually up to 750 °C. An inert atmosphere is maintained in the quartz tube. When the temperature exceeds one-half to two-thirds of melting temperature of the powder material, significant solid-state atomic diffusion takes place, followed by recrystallization and grain growth. As temperature keeps increasing, themolysis occurs which burns out the organic components such as the remaining binder, dispersant, etc. Fig. 4 (c) shows green sample with dies and Fig. 4 (d) shows brown sample.

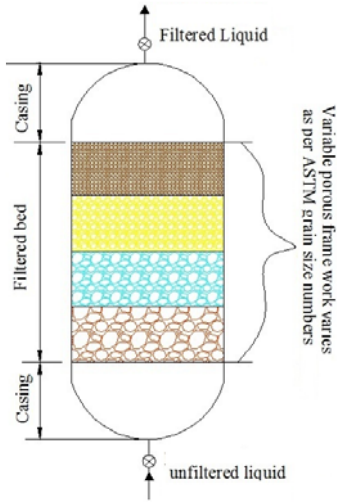

Fig. 5 Schematic diagram of filtration system

Fig. 5 shows the schematic diagram of filtration system to be implemented for the filtration purpose. The contaminated liquid filtration takes place from the bottom surface. The purified liquid will be flush out from the top surface. Fig. 6 shows the block diagram of liquid filter used for the filtration process. The filter is prepared by applying suitable binding pressure in PIM process.

III. Results

Fig. 7 (a) shows the segregated grinding waste collected from the various industries. Fig. 7 (b) shows SEM images which observed distributed iron flakes with other impurities. Fig. 7 (c) shows SEM images indicating sharp particle size measured by ImageJ software. Particle length of grinding waste is observed in between 0.120 to 0.977 micron shown in Table III. Grinding waste flakes are sharper at the ends which help to create the porosity in the area of filtered bed. Fig. 8 (Sample A) and (Sample B) shows the Energy-dispersive X-ray spectroscopy (EDS) test report of different elements in weight percentage presents in the grinding waste. Table IV shows that major element in sample A is iron oxide with 86.29 weight % followed by carbon. In Sample B, major element observed is iron oxide with 90.95 weight % followed by carbon. The high percentage iron oxide is used as a base metal. Table IV shows some other impurities in the grinding waste as, Si, Mg, S, Ca, Cr and Mn.

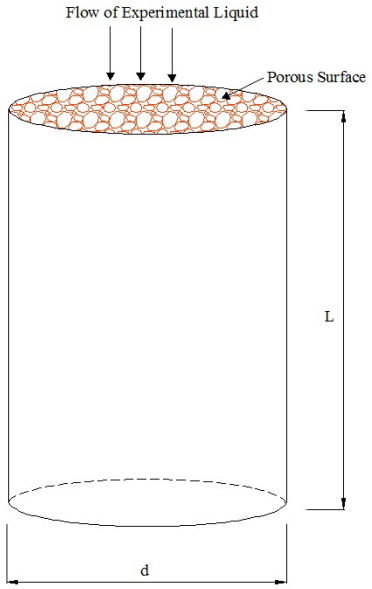

Fig. 6 Block diagram of liquid filter

TABLE III
Flake Size of Randomly Selected Grinding Waste Particles

Flake	Length	Flake	Length	Flake	Width
1	0.795	8	0.783	11	0.092
2	0.760	9	0.777	12	0.098
3	0.850	10	0.821	13	0.052
4	0.831	14	0.152	16	0.068
5	0.977	15	0.141	18	0.090
6	0.839	17	0.120	19	0.099
7	0.686	--	--	--	--

TABLE IV
Amount of Individual Elements Present in Grinding Waste

Elements	Weight	Elements	Weight
C	10.65	C	6.44
O	28.93	O	7.58
Mg	0.46	Cr	1.08
Si	0.32	Mn	1.53
S	0.53	Fe	83.37
Ca	0.58	--	--
Cr	0.38	--	--
Mn	0.78	--	--
Fe	57.36	--	--
Totals	100.00	Totals	100.00

Table V shows that solvent debinding process removes maximum wax added in the green sample as compared to

thermal debinding process. Fig. 9 shows the few % of wax is trapped inside the green sample. It may be further removed during the sintering process which is helpful for creating porosity in the sample and also improved the strength of samples with considerable shrinkage. Fig. 10 (A) shows SEM image with few sights agglomerated and more wax. Fig. 10 (B) shows SEM image with open pore after the removal of the wax which possess suitable porosity.

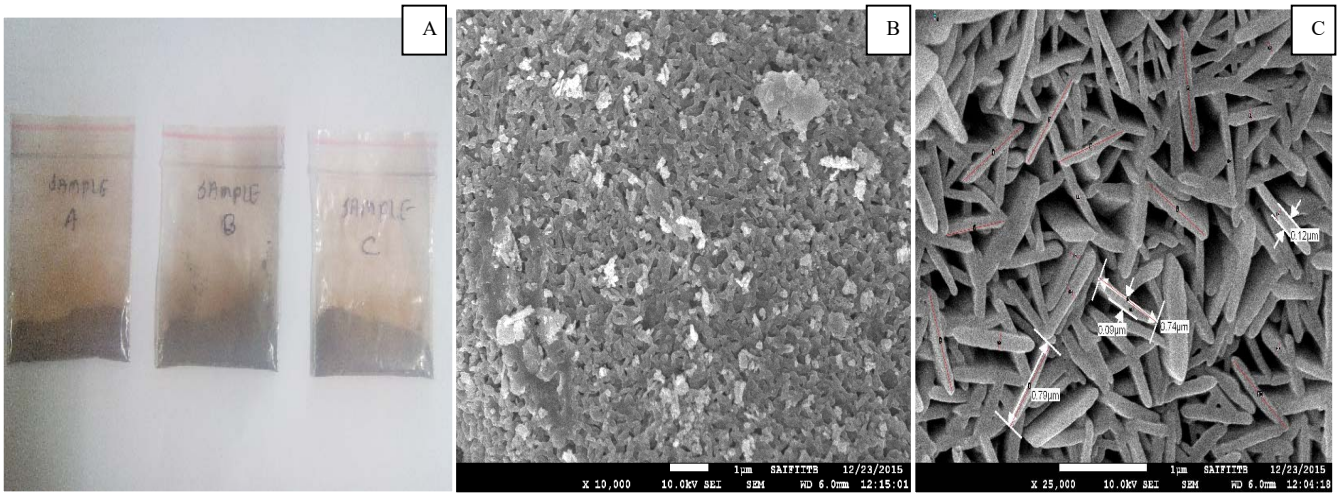

Fig. 7 (A) Grinding waste and (B) and (C) SEM images describing surface morphology and particle size (SAIF, IITB)

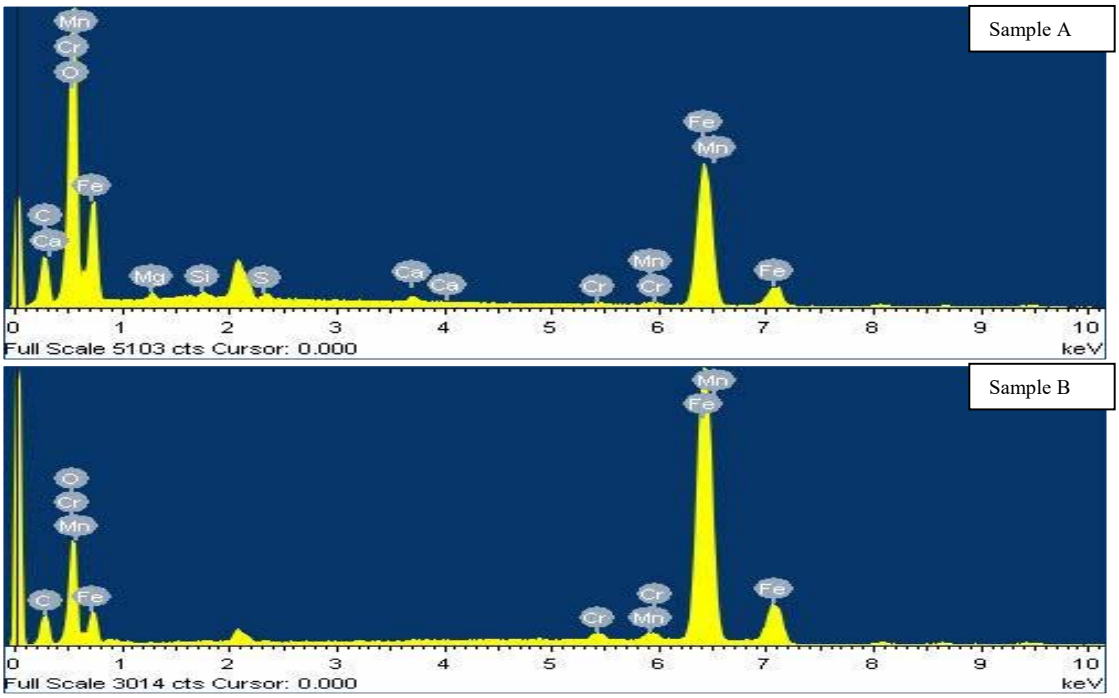

Fig. 8 Energy-dispersive X-ray spectroscopy (EDS) image of grinding waste of sample A and Sample B (SAIF, IITB)

TABLE V
ESTIMATION OF SOLVENT AND THERMAL DEBINDED SAMPLE D (91-9)

Target mass = Initial mass – (Initial mass *0.09*0.7) *Grinding waste						
Sr No.	Initial Mass (gms)	Target Mass	Solvent Debinding		Thermal debinding	
			Actual mass	% debinding	Actual mass	% debinding
D1	11.026	10.254	10.794	94.99	11.009	93.14
D2	3.925	3.650	3.477	104.97	3.857	94.63

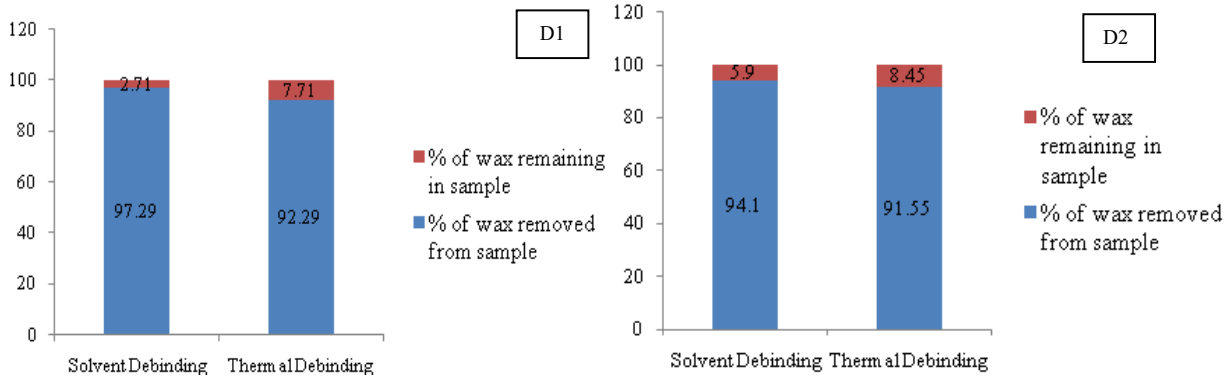

Fig. 9 Solvent and thermal debinded Sample D1 and D2

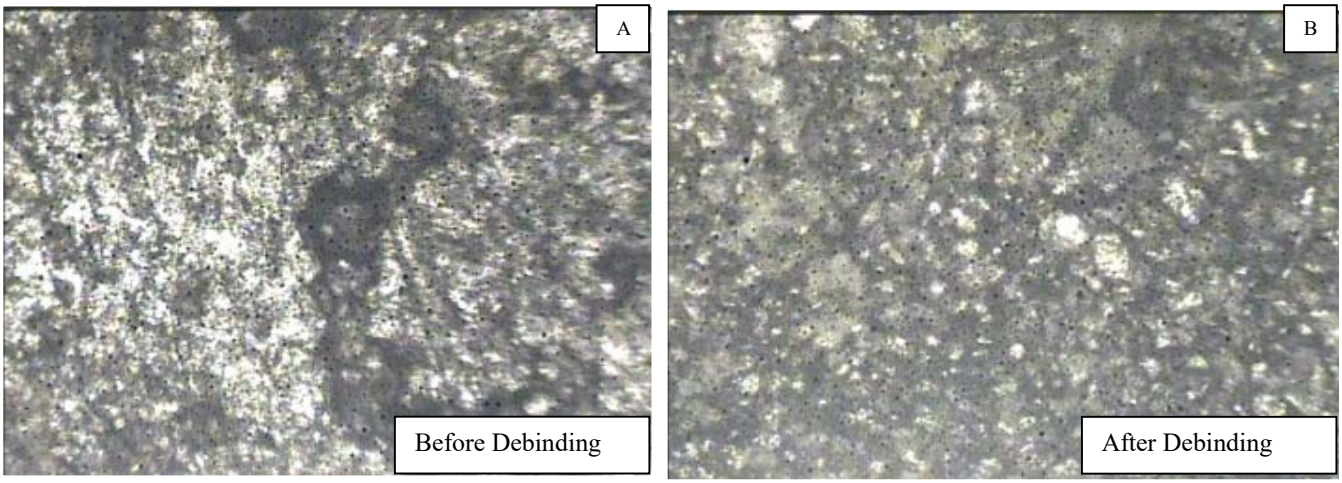

Fig. 10 (A) and (B) SEM Image of samples D (Before/After debinding)

IV. Conclusions

The variable porous samples are successfully produced by the PIM process. The SEM image shows that the grinding wastes obtained from the industry have a flake-like structure with 0.052 to 0.099 microns width and 0.120 to 0.977 micron length. Grinding waste flake is sharper at the ends which help to create the porosity in the area of filtered bed. Green mixtures contain the calloused kind of material which helps in creating the green samples. The solvent debinding suggests that the sufficient wax removed from the samples create porosity in the sample. The sintering improved the strength of samples with considerable shrinkage. FEG-SEM analysis suggests that few sites agglomerate but samples possess suitable porosity. Filters can be used from 2 years to 5 years without change because carbon nanotubes help to protect from damage and excessive wear. The various shapes are produced and they are under trial for various filtration tests as per IS3351-1968.

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