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Visible irradiation induced photodegradation by NbC/C nanocomposite derived from smoked cigarette litter (filters)

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A B S T R A C T

In a view to the treatment required to degrade organic effluents and also waste utilization, a non-treditional photocatalyst (NbC/C nanocomposite) has been synthesized by using Nb_2O_5 and cigarette litter (filter) at relatively low temperature 800 °C. With the help of XRD diffraction technique, the synthesis parameters have been optimized to get NbC/C nanocomposite and studied their morphology by transmission electron microscopy (TEM). The synthesized nanocomposite NbC/C was used to study the photocatalytic degradation of methylene blue (MB) dye under visible light illumination. Further, the effect of concentration of dye and photocatalyst has been studied under same conditions. With the help of BET, UV–visible absorbance, XPS, mass spectrometry and total organic carbon analysis, degradation mechanism has been established for the optimized nanocomposite sample.

1. Introduction

Nano-sized semi-conductors and doped semi-conductors such as TiO_2, ZnO, ZnS and TaN. are being used efficiently to degrade different types of organic pollutants and also for hydrogen production through photo-catalysis (Kaur et al., 2015; Liu et al., 2006; Mittal et al., 2014; Nurlaela et al., 2016; Rather et al., 2017). These pollutants, mostly dyes are the major byproducts of various industries like pharmaceutical industries, textile industries and paper-pulp industries, are directly disposed to natural water. Such polluted water is highly undesired due to the toxic ingredients causing cancer or human mutation (Zaharia et al., 2009). Moreover, coloration due to dyes can also cause hindrance to the sunlight and oxygen penetration affecting the aquatic living organisms (Meetani et al., 2011). These dyes are quite stable compounds, remain dissolved in water which are being removed by various treatment processes like reverse osmosis, ultra-filtration and adsorption. but, the generation of toxic byproducts reduced their applicability (Akyol and Bayramoğlu, 2005). Apart from water pollution, soil pollution due to the various polymeric litter/garbage also causes severe health hazards to society where degradability of solid waste is a critical issue.

Smokers contribute to air and soil pollution both, by exhaling smoke to air and throwing smoked cigarette on road sides, river banks and at sea beaches. Cigarette filters (CFs), a major left over component of smoked cigarette, are being thrown inappropriately at other places also. It is estimated that approximately 9 trillion filtered cigarette would be consumed by 2025 (Novotny et al., 2009; Shafey et al., 2009). CFs, a fibrous component consisting cellulose acetate as a major constituent, are photodegradable under the exposure of UV radiation in ideal environmental conditions but not biodegradable. Various studies have shown that there is an ambiguity on the biodegradability of cellulose acetate (Puls et al., 2011). Cellulose acetate is a very good source of carbon and can be used to develop various engineering materials such as transition metal carbides (TMCs) at relatively low temperature.

TMCs are considered as promising compounds for high temperature applications, tribology, cutting tool applications, fuel cell and corrosion applications (Fernandes et al., 2015; Liu et al., 2013; Nedfors et al., 2011; Rathod et al., 2011; Yate et al., 2015). Among all these, niobium carbide (NbC) is considered as a good catalyst support, grain growth inhibitor for Co-WC and alternate material for hard tissue implantation (Huang et al., 2008; Liu et al., 2013; Yate et al., 2015). It is also observed that NbC exhibits superconductivity below 11.5 K (Jha and Awana, 2012). Moreover, Chen et al. (2013) have reported the photodegradation of rhodamine B using NbC catalyst. However, its detailed study such as the effect of concentration of photocatalyst and dye solution has not been reported yet. For such diverse applications, synthesis of nano NbC at relatively low temperature becomes very critical due to higher specific surface area at nano scale. Generally, NbC has been synthesized at high temperatures (above 1000 °C) (Chen et al., 2015; Jha and Awana, 2012; Qiu et al., 2013; Song et al., 2015; Zou et al., 2011). In the present study, relatively low temperature synthesis of nano NbC/C nanocomposite using Nb_2O_5 as precursor of Nb and CF as carbon source has been reported along with its photocatalytic

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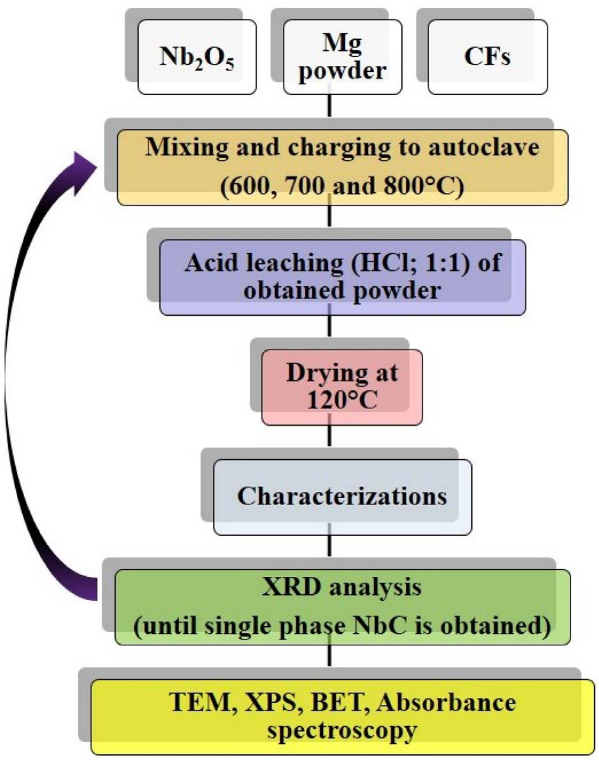

Fig. 1. Step-wise process showing optimization of the synthesis parameters to obtain nano NbC.

activity where degradation of organic pollutant (methylene blue; MB) under visible irradiations has been done.

2. Experimental procedure

2.1. Synthesis of NbC/C nanocomposite

Niobium pentoxide (Nb_2O_5), magnesium metal powder (Mg) and smoked cigarette filters (CFs) of Gold Flake, ITC Ltd. were taken as 1.329 g, 2 g and 1 g as niobium source, reducing agent and carbon source, respectively. The mixture of reactants was heated to different temperatures (600, 700 and 800 °C) at the rate of 5 °C/min for different holding time (5, 10, 15 and 20 h) in a stainless steel autoclave. Furthermore, the autoclave was cooled within the furnace to avoid the variability of cooling rate. The as synthesized black powder was leached with diluted HCl (1:1) and washed several times with distilled H_2O to remove acid content followed by drying at 120 °C. The synthesis methodology and parameters along with the sample details are represented and provided in Fig. 1 and Table 1, respectively.

Table 1
Details of synthesized samples.

Sample ID	Temperature (°C)	Holding time (h)
C-1	600	5
C-2	600	10
C-3	600	15
C-4	700	5
C-5	700	10
C-6	700	15
C-7	700	20
C-8	800	5
C-9	800	10
C-10	800	15
C-11	800	20

Table 2
Details of ICDD cards used for XRD analysis.

S. no.	Compound	Formula	ICDD card
1.	Niobium carbide	NbC	01-089-3690
2.	Niobium metal	Nb	01-089-3715
3.	Niobium oxide	NbO	00-042-1125
4.	Niobium dioxide	NbO_2	00-034-0898
5.	Niobium penta-oxide	Nb_2O_5	01-071-0005
6.	h-carbon	C	01-089-8487

2.2. Characterizations

Morphological features of cigarette filter were recorded with the help of *JEOL 6510LV (Japan)* accompanied with LaB_6 electron source (operated at 15 and 20 kV). To analyze the phase composition of as synthesized samples, X-ray diffraction (XRD) patterns were obtained by *PANalytical Xpert-Pro* diffractometer attached with Cu Kα radiation (1.5406 Å; Ni filter and step size = 0.013°) in the range of 20–80° (2θ). Diffraction peaks were then matched with ICDD standard cards on *X'pert High Score* as shown in Table 2. Further, the morphology of synthesized powder samples was recorded by transmission electron microscope (TEM) *JEOL 2100F,* operating at 200 kV. To analyze the specific surface area and pore distribution on the surface of the single phase NbC/C nanocomposite sample, *Microtrac Bell* was used to obtain N_2 adsorption-desorption behavior with pretreatment at 150 °C. Double beam UV–Visible spectrophotometer (*Hitachi U-3900H*) was used to obtain absorption spectrum of optimized sample. Further, surface elemental composition of photocatalyst has been analyzed by X-ray photoelectron spectroscopy (XPS) on PHI 5000 Versa Prob II, FEI Inc. with the help of Al Kα radiation (1486.7 eV, Ag standard).

2.3. Photocatalysis

For photocatalytic study of the optimized nanocomposite sample, aqueous solution of MB dye was used, this is followed by the addition of photocatalyst after achieving homogenous solution with continuous stirring. Thereafter, solution was stirred for 30 min in dark chamber to establish adsorption-desorption equilibrium, followed by the exposure of visible irradiation by CFL lamp (85 W; 8900 lx) with 1 h as sampling interval. Double beam UV–Visible spectrophotometer (*Hitachi U-3900H*) was used to observe the extent of reduction in MB concentration under visible irradiation at each sampling interval which was centrifuged at 4500 RPM to separate photocatalyst from dye solution. To study the effect of concentration of dye and photocatalyst different concentrations of dye (1, 2 and 3 mg/L) and photocatalyst (20, 40, 60 and 80 mg/L) were used. To confirm the diminishing absorbance with respect to irradiation time corresponding to degradation of MB dye, direct mass spectrometry (MS) and total organic carbon (TOC) were carried out on UPLC-XEVO-G2-XS/QTOF mass spectrometer and MULTI N/C 3100 (N3-800/O), respectively. Moreover, for the establishment of the mechanism of the degradation of MB dye was estimated by detecting the generation of reactive oxidative species in which separate photodegradation experiments adding different scavengers (1:10 volume ratio of 1 mM concentration) such as isopropanol (IPA, $^{\cdot}OH$ radical scavenger), sodium sulphate (SS, e^- scavenger), ammonium oxalate (AO, h^+ scavenger) and ascorbic acid (AA $^{\cdot}O_2^-$, radical scavenger) were performed.

3. Results and discussion

3.1. SEM analysis of cigarette filters (CF)

Fig. 2 shows the typical structural features of CFs having triangular cross section of fibers. Each cigarette filter consists of long continuous fibers, entangled to filter by-products of burned cigarette (tar, nicotine