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GREEN SYNTHESIS OF $NiWO_4$ NANOPARTICLES USING PHYLLANTHUS AMARUS EXTRACT

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ABSTRACT

Synthesis of nanoparticle by green method has attracted much attention due to its application in various fields. In the present study nickel tungstate nanoparticles were synthesized using the plant extract of phyllanthus amarus (kelanelli). The synthesised $NiWO_4$ nanoparticles were characterized by Fourier transform infrared spectroscopy **(**FT-IR), X-ray diffraction (XRD) and Scanning Electron microscopy (SEM). XRD results revealed that the average crystallite size of $NiWO_4$ nanoparticle was 33.49 nm. SEM results showed that $NiWO_4$ nanoparticles composed of flower in shape. The bands appeared in FT-IR Spectrum at 750cm^{-1} to 1000cm^{-1} corresponds to (W-O) stretching of WO_4. The antimicrobial activities of synthesized nanoparticles were studied against various pathogens.

Keywords: $NiWO_4$, Phyllanthus amarus, IR, XRD, Antimicrobial activity

I INTRODUCTION

Metal tungstate with formula MWO_4 have attracted much attention due to their interesting size and shape dependent optical, magnetic and electronic properties [1 -5]. Transition metal tungstate have many applications, such as in gas sensors, optical fibers, humidity sensors, pigments, catalytic and biological activity. Nickel tungstate ($NiWO_4$) is one of the important inorganic materials that have high application potential in various fields such as catalysis, humidity sensor, photo anodes, laser hosts, microwave application and optical fibers [6-8]. Also it is considered to be a potential candidate as a visible light photo catalyst for organic contaminants decomposition. Recently, many studies have been reported on the preparation and characterization of nickel tungstate using various method such as Czochralski method [9], precipitation [10], hydrothermal [11], solid state [1], pulsed laser deposition [12]. However, these methods usually require special equipment, high temperature, templates or substrate and usually result in impurities. The toxic chemicals, non polar solvent and synthesis additives or capping agent immersing from the chemical reaction are often potentially dangerous to the environment.

The plants with the antioxidant or reducing properties are usually responsible for the reduction of metal compounds into their respective nanoparticles. According to the researchers, the poly-ol components present in the plant extract are responsible for the reduction of metal ions, where as water soluble heterocyclic components stabilize the formed

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nanoparticles. The synthesized nanoparticles showed considerable stability and it was found to have potential for bio-application.

Gupta *et al* studied adsorption removal of methylene blue by guar gum-cerium (IV) tungstate as a hybrid cationic exchanger [13]. Zawawi *et al* synthesized metal tungstate by sucrose template method to study its structural and optical properties[14]. The synthesis of nano-size $NiWO_4$ crystals includes co-precipitation, polymeric precursor method, modified citrate complex technique, hydrothermal method, molten salt method and spray pyrolysis[15,16].

Hence developing of reliable biosynthetic, an environment friendly approach has added much importance because of its eco friendly products, biocompatitility and economic viability in the long run and also to avoid adverse effects during their environment application. In this work, we adopted a precipitation route for the synthesis of $NiWO_4$ pure phase with uniform particle size distribution and plant extract as capping agent. The antimicrobial activity of the prepared nano photo catalyst was investigated. $NiWO_4$ nanoparticles were explored with respect to their prospective antibacterial application.

II EXPERIMENTAL

2.1 Preparation of Plant Extract

The collected *phyllantus amarus* leaves were washed thrice with tap water and twice with distilled water to remove the adhering salts and other associated contaminants. 10g of leaf was taken and boiled with 100 ml of sterile distilled water at 100^0 C for half an hour. The boiled extract was filtered through what man No.1 and was stored in refrigerator at 4^0 C for further studies[17].

Fig.1 Phyllantus amarus leafs

2.2 Preparation of $NiWO_4$ Nanoparticles

$NiWO_4$ nanoparticles were prepared by simple precipitation method. 0.5M $NiCl_2$ and0.5 M of $NaWO_4.2H_2O$ was dissolved separately in 50 ml distilled water in two glass beakers. $NaWO_4$ $.2H_2O$ was added to $NiCl_4$ solution drop wise with constant stirring while led to the rapid formation of green color precipitation. To this 5ml of plant extract was added and it was stirred for 2 hours to form a homogeneous precipitation. After filtering, the precipitate was

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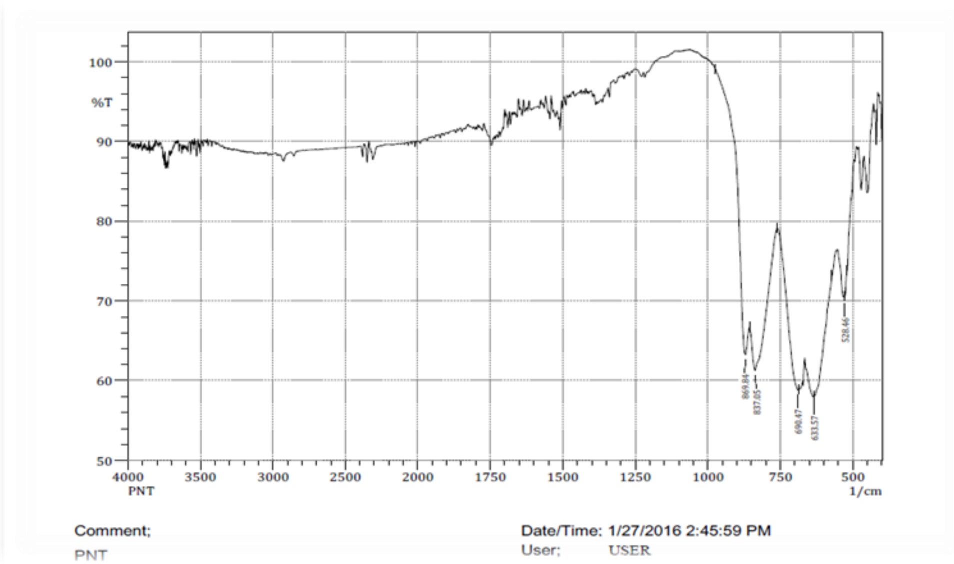
washed with distilled water and dried at 80^0 C for 5 hour. The obtained nano particle was calcinated at 500^{0}C for 1 hour [18].

III RESULTS AND DISCUSSION

3.1 FT- IR

The FT-IR spectra of NT and PNT were shown in fig.2. It has been reported that the main absorption bands of $NiWO_4$ appeared in the range of 450 – 1000 cm^{-1}. The bands appeared at 878 and 824 cm^{-1} arise from vibration of the WO_2 entity present in the W_2O_8 groups. The absorption band at 629 cm^{-1} was typical of a two oxygen bridge (W_2O_2) and corresponded to the asymmetric stretching of the same unit. Additionally the observed band at 450 cm^{-1} confirms the stretching vibration of the NiO_6 polyhedral building NiO validating the formation of $NiWO_4$ structure. The peaks at 1120 cm^{-1}, 1630 cm^{-1}, 2360 cm^{-1} and 3442 cm^{-1} represent the diverse functional groups of the absorbed biomolecules on the surface of the $NiWO_4$ particles. The peaks around 1120 cm^{-1} denoted stretching vibrational bands responsible for compounds like flavonoids and terpenoids. Peak at 1465 cm^{-1} corresponds to the C-N Stretching mode of aromatic amine group. The band at 1643 cm^{-1} can be assigned to the amide I band of the proteins and aromatic ring, 2360 cm^{-1} confirmed to C-C and 3442 cm^{-1} was related to N-H stretching [19]. The variation in the position indicated presumably some metabolites such as tannins, flavonoids, alkaloids and cartenoids which are abundant in plant extract. The shift in the peak position is due to its surface modification.



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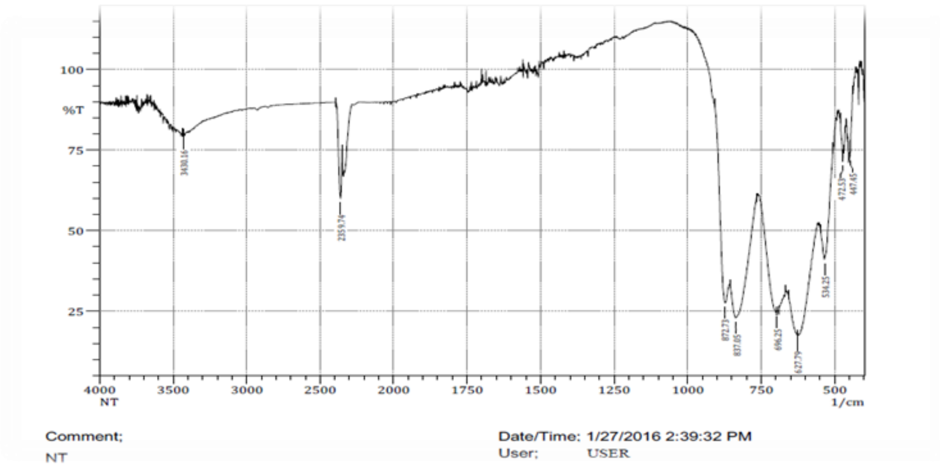

Fig.2. FT-IR spectra of PNT and NT

3.2 XRD

XRD is used to investigate the changes of phase structure and crystallite size of the synthesized nanoparticles with and without addition of plant extract. Fig.3. Shows the XRD pattern of NT and PNT. The main diffraction peaks of PNT with monoclinic phases at 19.3^0 , 23.9^0 ,24.95^0 ,30.9^0 ,36.5^0 ,41.6^0 ,54.6^0 and 65.6^0 (JCPDS-150755) which corresponds to (100), (100), (110), (111), (002), (102), (202) and (132) crystallographic planes respectively and is indexed as the primitive lattice. It can be see that $NiWO_4$ nanoparticles embedded in plant extract XRD peaks were broad when compared with the nanoparticles without plant extract. The average crystallite size was calculated using the Debye's Scherer equation [20].

$$D = K\lambda / \beta \cos\Theta$$

Where, β is the full width at half height maximum of the most intense 2Θ peak, K is the shape factor(0.89). Θ, λ are the incident angle and wavelength of X-rays respectively, The average particle obtained for NT nanoparticle is 48.72 nm while the size was found to reduced to 33.49 nm for PNT.

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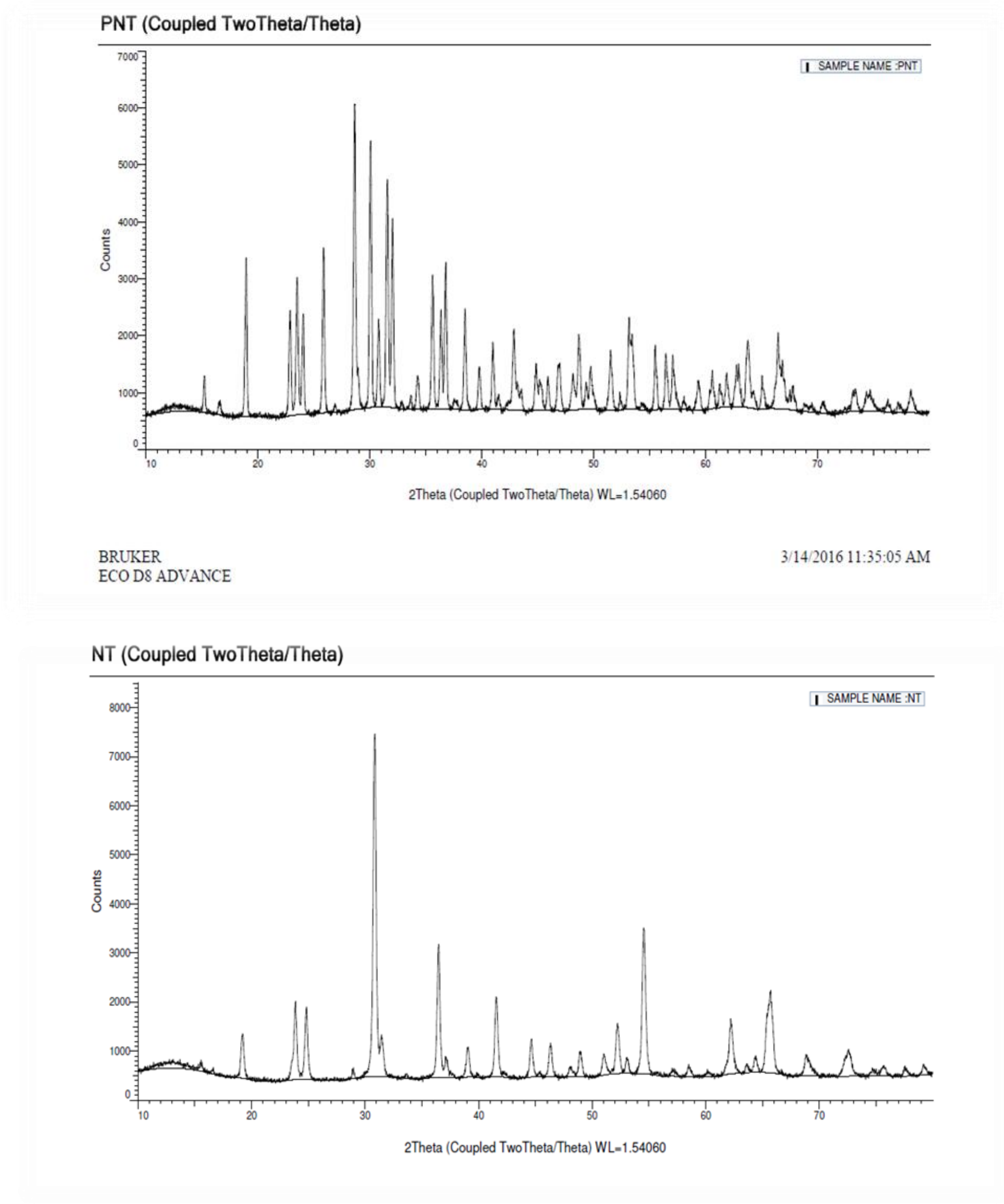

Fig 3. XRD pattern of PNT and NT

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3.3 SEM

SEM images of the synthesized $NiWO_4$ were shown in the Fig 4.a&b. After the addition of plant extract the surface modification takes place and $NiWO_4$ crystals with certain faces continue to grow into a more stable structure which makes the product present a small flower shaped structure with uniform diameter [21].

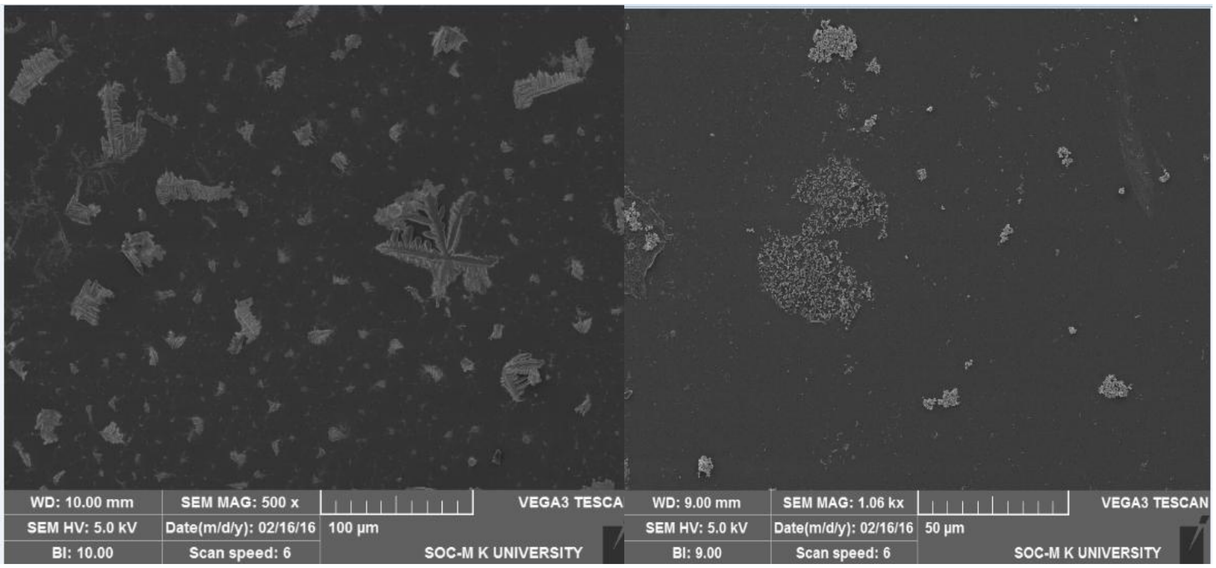

Fig 4 (a). SEM image of NT

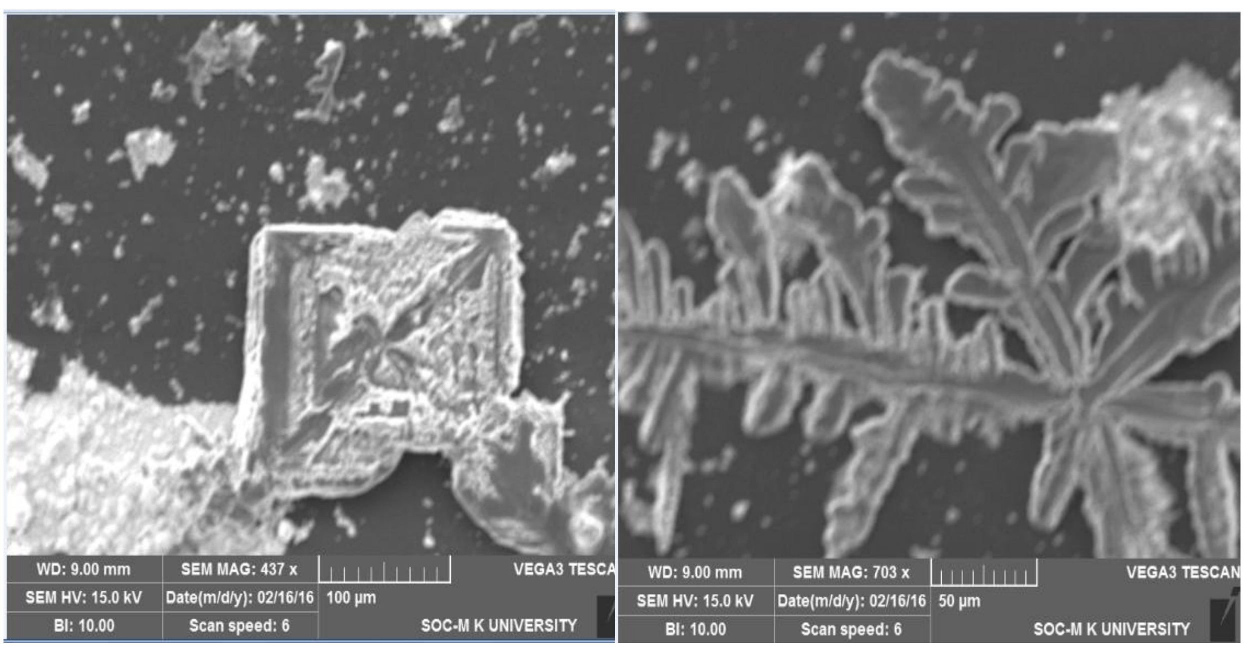

Fig 4(b) SEM image of PNT

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3.4 Antimicrobial Activity

The antimicrobial activity of the NT nanoparticles was tested against the gram positive (Staphylococcus aureus), gram negative (Salmonella) and fungal species (Aspergillusniger). The results for the antimicrobial activity of NT and PNT are shown in below table and it is observed that PNT showed excellent anti bacterial activity against staphylococcus aureus and Klebsiella. Salmonella is resistance in the presence of NT and PNT shows excellent activity against this pathogens. NT shows excellent activity against Aspergillus Niger shows resistant against PNT. The remarkable antimicrobial activity of NT and PNT are due to the generation of surface oxygen species which leads to the pathogens [22].

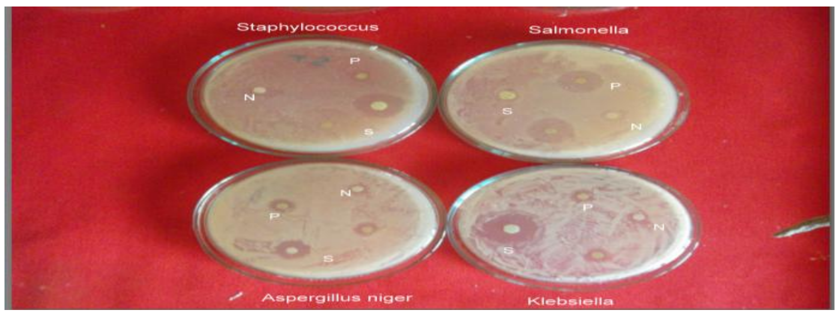

Fig .5. Antimicrobial activity of PNT& NT

Table 1 Antimicrobial activity of PNT& NT

Organisms	Concentration of extract and zone inhibition(mm)	
	PNT(100µl)	NT(100µ)
Staphylococcus aureus	19	18
Salmonella	22	21
Aspergillus niger	24	23
Klebsiella	18	25

IV CONCLUSION

The Nickel tungstate nanoparticles were successfully synthesized by using *Phyllanthus amarus* plant to use antimicrobial study which provides cost effective, easy and proficient way for synthesis of $NiWO_4$ nanoparticles. The functional group present in leaf extract has been confirmed by FT-IR. The functional group of leaf extract were mainly responsible for the reduction of Nickel tungstate metal ion into nanoparticles.The synthesized Nickel

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tungstate nanoparticles were analyzed using FT-IR, XRD, and SEM. Nickel tungstate nanoparticles were effective utilized for the antimicrobial activity study. The maximum ZOI was found to be more in gram negative bacteria when compared to gram positive bacteria. The *phyllanthus amarus* plant may be effectively utilized for the production of Nickel tungstate nanoparticles with economically for many pharmaceutical applications.

REFERENCES

1.C.W.E.van Eijk, Nucl. Instrum. Meth A (1997)285-290

2.R.Sundaram, K.S. Nagaraja, Meter.Res.Bull.39(2004)557-565

3.H.Wang, F.D.Medina, Y.D.Zhou,Q.N.Zhang, Phys.Rev.B 45 (1992) 10356-10361

4.J.M Quintana-Melgoza, A.Gomez-Cortes, M.Avalos-Borja, React. Kinet. Catal. Lett. 76(2002) 131-140.

5.B.Seheffer, P.Molhoek, J.A.Moulijn,Appl. Catal. 46(1989) 11-19

6.T. Rabizadeh,S.R. Allahkaram, Master. Design.31(2010) 3174-3179

7.J.HO Ryu , J.Yoon, C.S. Lim, W. Oh,Shim,Ceram. Int.31(2005) 883-888

8.R.C.Pullar, S.Farrah,N.McN. Alford,J.Eur. Ceram.Soc.27(2007) 1059-1063

9.D.L Stern, R.K. Grasselli, J. Catal.167(1997) 570-572.

10.R.Sundaram. K.S. Nagaraja, Master . Res. Bull. 39(2004) 581-590.

11.P.S.Pandey,N.S.bhave,R.B.Kharat, Electrochim. Acta 51(2006) 4659-4664.

12.M.Asif,Journal of Pharmaco.Phytochem 4(2012) 78-83.

13. V.K.Gupta, D.Pathaniab,P.Singh, A. Kumar, B.S.Rathore, carbohyd. Polym101 (2014).684-691

14.M.Zawawi, R.Yahya, A.Hassan, H.N.M. Ekramul Mahmud, M.N.Daud, Chem. Cent.J(2013).80

15.A.L.M. de Oliveira, J.M. Ferreira, M.R.S silva, G.S. Braga, L.E.B. Soledade, M.A.M.A.Maurera, Dyes pigments (2008).210-216

16.A.Sen, P.A.Pramanik, J, Eur. Ceram. Soc. 21 (2001).745-753

17.R. Karimiana, F.Pirib, J. Nanostructure 3(2013) 87-92

18.S.M.Pourmortazavi, M.R.Nasrabadi, M.K. Shalamzari, M.M.Zahedi, S.S.Hajimirsadeghi, I.Omrani, Appl. Surf. Sci (2012)745-752

19.P.V.Kamat, D.Meise, Curr Opin Collaid Interface Sci.7(2002)282-287

20.M.Nasir, S.Bagwasi, Y.Jiao,F.Chem,B.Tian, J.Zhang, Chem. Eng. J. 236(2014)388-392

21.B.Subhash, B.Krishnakumar, V.Pandian, M.Swaminathan, M.Shanthi, Sep. Purif. Technol. 96(2012)204-213

22.N.Riaz, F.K.Chong, B.K.Dutta, Z.B.Man. M.S.Khan, E.Nurlaela, Chem. Eng .J.108 (2012)185-186