SYNTHESIS AND CHARACTERIZATION OF NICKEL OXIDE NANOPARTICLES FROM FISH SCALE EXTRACT OF SILVER CARP, *HYPOPHTHALMICHTHYS MOLITRIX*

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Abstract :

The characteristics of biosynthesis methods, such as safety, low cost, and environmental friendliness are deemed attractive by researchers. Green synthesis method was employed in this study to synthesize from biological sources. Green synthesis of NiO NPs is environment friendly and low cost methods to develop the nanoparticles. A unique fish scale powder enriched with collagen was used for the preparation of NPs. The difference in densities of fish scale constituents, i.e., collagen and calcium/magnesium phosphate was the main reason for the possibility of the separation of the ground cotton-like product into two parts: powder enriched with inorganic components and fluff enriched with organic (collagen) components. Collagen helps in binding with the Nickel. In the resulting procedure Ni(0) gets oxidized by calcinations to produce copper oxide nanoparticle (NiO NPs) by heating in furnace. . The use of XRD resulted in successful synthesis and categorization of NiO NPs. The morphology and particle size 31.52nm were also investigated.

Introduction :

The field of Nanotechnology flourished notably in the last twenty years, and this help scientists explore more and more fields of applications. Nanotechnology is the science of making, designing, and their use as nanomaterials and the study of the relationship between various properties of materials and their nanometer dimensions (Siddiqi and Husen, 2017). Nanoscience is a scientific technique to synthesize particles in the nanoscale range, from 1 to 100 nm (Mittal *et al.*, 2013). Nanoparticles (NPs) are gaining considerable attention in the scientific society and industries due to their remarkable perspective on the future. The synthesis of metal nanoparticles has obtained significant attention in last 2 decades due to their uncommon possessions and potential applications in different applied fields (Mobasser and Firoozi 2016). Nickel oxide nanoparticles (NiO-NPs) possess a number of particular characteristics and it is widely used in batteries, printing inks, diesel-fuel preservative, electrochromic films, magnetic materials and also act as a catalyzer in many biochemical reactions (Salimi *et al*., 2007 and Schrand *et al*., 2010). The biological synthesis of metallic NPs is becoming an essential research interest for interdisciplinary scientists around the world as the conventional chemical methods require more energy and reagents as well as harmful and toxic, too for consumption, compared to biological methods. Moreover, the literature on this field is very meager, and the synthesis protocol often employs the use of expensive, harsh, and toxic chemicals (Sannegowda *et al.* 2014). Therefore, in this pursuit, the development of new green chemistry synthetic procedures using environment-friendly materials, non-toxic

solvents, and reagents is highly required with minimal wastage in terms of raw materials and energy. In this aspect, we wish to report the utilization of the fish scale of silver carp, a common fish market waste material for the preparation of NiO-NPs. The fish scale compositional abundance of type I collagen fibers (41–84 %) has been reported (Ikoma et al. 2003). These components may be predicted for the effective reduction and stabilization of NiO-NPs generated using fish scale extract by a greener route.

The carp nanaoparticles are between 1 nm and 100 nm in size has many applications due to large degree of commercialization. It is an attractive material for its distinctive properties, such as good conductivity, chemical stability. Silver carp (*Hypophthalmichthys molitrix*) is economically important fish with largest reported production in aquaculture globally. It is native to eastern Asia, and is a large herbivorous freshwater fish of family Cyprinidae. Antioxidant activity of collagen peptides from Oreochromis sp. scales, and the results showed that collagen peptides with a molecular weight of 3–10 kDa had the highest antioxidant activity (Sierra *et al.* 2021). This collagen rich scale from silver carp will stick to the nickel and play important role to development of the nickel nanoparticles. There is very less information on the bio-availability and effect of nickel oxide nanoparticle (NiO NPs) in silver carp (*Hypophthalmichthys molitrix*). Therefore, the objective of the study was to develop green synthetic NPs and its characterization.

The present project was planned to synthesize nickel nanoparticles (NiO NPs) from scales of Silver carp, *Hypophtalmichthys molitrix* and its characterization of NPs by using XRD.

Materials and Methods :

Fig-1 Photograph of Silver carp *Hypophthalmichthys molitrix*

Fish scales from Silver carp, *Hypophthalmichthys molitrix* were collected from local fish market in Nagpur region. The fish scales were washed with water to remove dirt and other impurities and then fish scale samples were sun dried for 3 days and final dried in oven at 80°C., grind the sample and used for the synthesis of NiO NPs. To take 10g of fish scale powder was mixed with 100ml of distilled water, then boiled water bath at 70°C in 20 minutes. After cooling few minutes, sample was filter by using Whattman filter paper obtain the fish scale extract. Finally, the plain extract was preserved at 4 ˚C.

Biogenic synthesis of NiO NPs :

In order to prepare the NiO NPs, 10 mL of the extract was mixed to 100 mL 0.1N $NiSO_4$ solution and the mixture was heated at 70 °C for 60 min. Incubate at dark condition for 72 hrs. After, 3 days sedimentation formed. The calcinations of sediment powder done at 550 °*C* for 5 hrs in the muffle furnace(*NiO NPs*).

The chemical structure of the products was characterized by XRD. Meanwhile, the NiO NPs particle size was determined using the Debye Scherrer equation,

$D=K\lambda/\beta Cos\theta$

Where D is the nanoparticle crystalline size, K represent the Scherrer constant (0.98), λ denotes the wavelength (1.54), β denotes the fullwidth at half maximum (FWHM).

Fig- 2. Photograph of scales of Silver carp, *Hypophthalmichthys molitrix*

Results and Discussion :

Biological source was prepared because of its biological safety and low cost. NiONPs were synthesized from fish scale extract of Silver carp, *Hypophthalmichthys molitrix* (Fig. 1). We developed a trouble-free protocol for synthesis and characterization of NiONPs from the fish scale extract of Silver carp, *Hypophthalmichthys molitrix*. About 10% of the fish scale *extract was mixed to 100 mL $NiSO_4$* (0.1N) *solution and the mixture was heated at 70 °C for 100 min.* Incubate at dark condition for 72 hrs. After, 3 days sedimentation formed. The calcinations of sediment powder synthesised at 550 °*C* for 5 hrs in the furnace (*NiO NPs*) to remove organic component. XRD analysis indicated that nickel nanoparticles are in noncrystalline form and has porous structure which is suitable for adsorption. The samples calcined at 700 °C show diffraction peaks characteristic of the cubic crystalline structure of pure NiO (JCPDS – 47–1049), with (111), (200), (220), and (311) as main diffraction peaks (Oh *et al.*, 2019)

XRD Analysis of CuONPs :

Crystallinity of the green prepared NiO NPs was determined by XRD study (Fig. 3). The observed diffraction sharp peaks position at 2θ = 15.550°, 17.43(°, 20.192°, 20.81°, 21.454°, 24.127°, 27.11°, 28.599°, 29.338°, 30.060°, 31.68°, 32.713°, 33.981°, 37.322°,

43.287°, 45.01°, 45.953°, 46.98°, 49.80°, 53.40°, 55.04°, 57.59° ,59.25°, 60.020°, 60.928°, 63.101°, 64.053°, 66.42°, 70.19°, 73.81°and 75.475° were assigned highly consistent in the present result and similar sharp peaks pattern reported in NiO NPs using crystal phases of rhombohedral (JCPDS card No. 89-3080) for pure NiO (Mir *et al.*, 2023). The estimated crystal size of NiO nanoparticles as calculated by Scherrer equation was 31.52nm

The XRD spectrum is shown in Fig. 3. Three main peaks with high intensity were observed in XRD at 2θ values of 28.599°, 33.981°, and 43.287°, shown in Fig.4. This result is equivalent to the higher peak intensity reported in previous studies (Mir *et al.*, 2023).

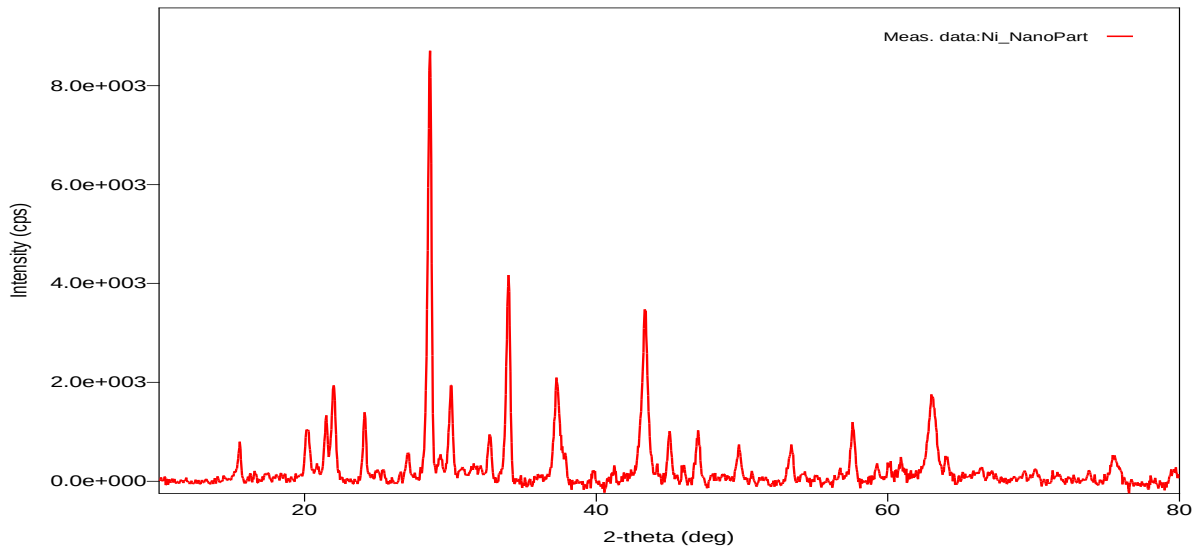

Fig. 3 Powder XRD pattern of biogenic NiO NPs.

Peak list :

No.	2-theta(deg)	d(ang.)	Height(cps)	FWHM(deg)	Int. I(cps deg)	Int. W(deg)	Size(ang.)
1	15.550(17	5.694(6)	514(33)	0.22(2)	177(10)	0.34(4)	379(41)
2	17.43(14)	5.08(4)	82(13)	0.7(3)	124(22)	1.5(5)	118(45)
3	20.192(9)	4.3941(19	732(39)	0.351(13)	326(10)	0.44(4)	240(9)
4	20.81(2)	4.264(4)	150(18)	0.32(6)	61(9)	0.41(11)	261(47)
5	21.454(7)	4.1385(13	808(41)	0.246(12)	252(9)	0.31(3)	343(17)
6	21.985(8)	4.0398(14	1304(52)	0.321(9)	531(14)	0.41(3)	263(7)
7	24.127(19	3.686(3)	945(44)	0.231(18)	269(14)	0.28(3)	367(29)
8	27.11(5)	3.287(6)	386(28)	0.26(4)	117(16)	0.30(6)	331(48)
9	28.599(4)	3.1188(4)	5976(112	0.282(3)	1988(20)	0.333(10)	304(3)
10	29.338(16	3.0419(16	284(24)	0.33(5)	112(13)	0.40(8)	256(36)
11	30.060(5)	2.9704(5)	1267(51)	0.287(10)	429(14)	0.34(2)	299(10)
12	31.68(7)	2.822(6)	148(18)	1.8(2)	314(33)	2.1(5)	48(7)
13	32.713(7)	2.7353(5)	655(37)	0.242(18)	187(12)	0.29(3)	357(27)
14	33.981(6)	2.6360(5)	2856(77)	0.289(5)	948(15)	0.332(14)	300(5)
15	37.322(14	2.4074(9)	1407(54)	0.413(19)	841(29)	0.60(4)	212(10)
16	43.287(4)	2.08847(1	2608(74)	0.333(12)	1419(20)	0.54(2)	268(10)

17	45.01(3)	2.0125(13	692(38)	0.26(2)	204(16)	0.29(4)	343(30)
18	45.953(19	1.9733(8)	225(22)	0.17(6)	42(12)	0.19(7)	516(180)
19	46.98(3)	1.9327(12	661(37)	0.31(2)	218(17)	0.33(4)	292(22)
20	49.80(4)	1.8295(14	517(33)	0.28(5)	203(15)	0.39(5)	332(55)
21	53.40(4)	1.7143(12	485(32)	0.32(4)	169(18)	0.35(6)	289(32)
22	55.04(4)	1.6670(11	93(14)	0.16(10)	15(10)	0.17(13)	601(378)
23	57.59(3)	1.5992(7)	876(43)	0.27(3)	317(17)	0.36(4)	346(41)
24	59.25(4)	1.5583(9)	266(24)	0.20(5)	65(10)	0.25(6)	468(110)
25	60.020(12	1.5401(3)	335(26)	0.16(3)	80(10)	0.24(5)	603(130)
26	60.928(13	1.5193(3)	365(28)	0.16(5)	120(13)	0.33(6)	586(184)
27	63.101(10	1.4721(2)	1282(52)	0.500(18)	986(21)	0.77(5)	195(7)
28	64.053(11	1.4525(2)	368(28)	0.18(4)	100(12)	0.27(5)	552(126)
29	66.42(6)	1.4064(10	133(17)	1.03(15)	212(21)	1.6(4)	96(14)
30	70.19(7)	1.3398(12	156(18)	0.47(16)	144(19)	0.9(2)	215(72)
31	73.81(3)	1.2828(4)	155(18)	0.23(6)	39(10)	0.25(10)	455(125)
32	75.475(17	1.2586(2)	342(27)	0.58(5)	211(22)	0.62(11)	181(16)

Fig.4 Peak list of XRD pattern of biogenic NiO NPs.

Some low-intensity peaks that appeared in the XRD pattern were observed with 2θ values are 15.550°, 17.43(°, 20.192°, 20.81°, 21.454°, 24.127°, 27.11°, 29.338°, 30.060°, 31.68°, 32.713°, 37.322°, 45.01°, 45.953°, 46.98°, 49.80°, 53.40°, 55.04°, 57.59° ,59.25°, 60.020°, 60.928°, 63.101°, 64.053°, 66.42°, 70.19°, 73.81°, 75.475° . 31.52nm Particle Size of NiONPs. The filler particle size often affects the mechanical properties of composites. Hence the characterization of filler size and their distribution will help to understand the properties of composite.

Conclusion :

The characteristics of biosynthesis methods, such as safety, low cost, and environmental friendliness are deemed attractive by researchers. Green synthesis method was employed in this study to synthesize from biological sources Green synthesis of NiO NPs is environment friendly and low cost methods to develop the nanoparticles. A unique fish scale powder enriched with collagen was used for the preparation of NPs. The difference in densities of fish scale constituents, i.e., collagen and calcium/magnesium phosphate was the main reason for the possibility of the separation of the ground cotton-like product into two parts: powder enriched with inorganic components and fluff enriched with organic (collagen) components. Collagen helps in binding with the Nickel. In the resulting procedure Ni(0) gets oxidized by calcinations to produce copper oxide nanoparticle (NiO NPs) by heating in furnace. The use of XRD resulted in successful synthesis and categorization of NiO NPs. The morphology and particle size 31.52nm were also investigated.

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