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GREEN SYNTHESIS OF IRON OXIDE NANOPARTICLES FROM Andrographis paniculata AND ITS APPLICATION IN SEPARATION OF OIL-SPILLS FROM WATER

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Abstract: The synthesis of particles which has a size within the range of 1-100 nm is known as "Nanotechnology". Nanotechnology is based on the concept of smaller the size, larger is the surface area and hence nanotechnology is a widely studied subject from many years. In the current project, Andrographis paniculata is used as the source for the synthesis of iron oxide nanoparticles. The synthesized iron oxide nanoparticles were characterized by UV spectrometry, XRD, FTIR, SEM and TEM. The results displayed that the nanoparticles had an absorption peak at 260 nm, spherical shape with an average size below 50 nm. The synthesized nanoparticles were employed in the separation of oil spill from water with the help of ferrofluid (small scale). Increase in the amount of ferrofluid enabled an efficient separation of oil spill from water, thus aiding in reduction of one of the major causes of water pollution in oceans..

***Index Terms* - Iron Oxide Nanoparticles, *Andrographis paniculata*, Green Synthesis, Characterization, Ferrofluid, Oil-Spill Water Separation.**

I. INTRODUCTION

As a widely studied subject, "Nanotechnology" can be applied in various fields of study such as; Agriculture, Textile, Environment etc. Physical, Biological and Chemical methods can be used for the synthesis of nanoparticles. Synthesis by chemical method involves harmful synthetic concoctions, the arrangement of perilous side-effects, and sullying from compound antecedents [1]. Microscopic organisms, algae, fungi and plant concentrates can be utilized in current choices for the synthesis of metal/metal oxide nanoparticles. Green-combined nanoparticles are related with natural substances from plant extract that improve molecule stability. Contrasted with microorganisms, plant-interceded nanoparticles are modest [2].

Andrographis paniculata, a medicinal plant which grows upto an height of 60-70 cm is also known as "Kalmegh". It is an annual and branched plant with lanceolate green leaves [3]. It grows in large numbers in India, Sri Lanka, Pakistan, Java, Malaysia and Indonesia. In India, it grows in the plains. *Andrographis paniculata* is also known as "King of Bitters" because of its bitterness [4-5]. The leaves and the stem of the plant is used to extract the phytochemicals [5]. The plant has various medicinal properties such as : Hepatoprotective, Anti-microbial, Anti-oxidant, Antipyretic, Anti-inflammatory, etc. The presence of Andrographolide and Neoandrographolide which are the active components contributes to the medicinal value of the plant. Both of these active components are derivatives of diterpenoids. The plant contains steroids, flavonoids, phenols, terpenoids, saponins, terpenoids which reduces the ionic metal to mass metallic nanoparticles [6].

Being present in more than one crystal structure and different structural and magnetic properties, iron oxide, a mineral compound is present abundantly in nature [7-8]. Hematite, Magnetite and Maghemite are the main forms of this mineral [8,9]. Crystal structure of the three oxides is explained as closely packed plains of oxygen anions with iron cations in octahedral interstitial sites [7,9 10-11].

Ferrofluid comprises of nanoparticles suspended in a bearer liquid. In this study, the capacity of the ferrofluid to move a specific way within the sight of an external magnetic field is applied in the partition of oil spill from water.

Fig 1: *Andrographis paniculata* leaves

II. EXPERIMENTAL

2.1 Preparation of *A. paniculata* leaf extract

About 100 grams of *A. paniculata* leaves were collected, shade dried and powdered using a kitchen blender. The powdered mixture was soaked in 300ml of double distilled water overnight at 4°C. After 24 hours, the mixture was boiled for 10 minutes and the extract was cooled to room temperature and then filtered using Whatman filter paper (No. 42) [12].

2.2 Synthesis of iron oxide nanoparticles using *A. paniculata* leaf extract

$FeCl_3.6H_2O$ and $FeCl_2.4H_2O$ were measured in 1:2 molar ratios and dissolved in 200ml of double distilled water and heated at 80°C with mild stirring using a magnetic stirrer under atmospheric pressure. After 10 minutes, 40ml of the leaf extract was added (color change to dark brownish color). After 10 minutes, 20ml of aqueous NaOH solution was added to the mixture at a rate of 3ml per minute which allowed iron oxide to precipitate consistently. The mixture was then cooled down to room temperature. The iron oxide nanoparticles obtained by decantation was subjected to centrifugation at 10,000 rpm for 10 minutes. The magnetites formed were washed with double distilled water and ethanol for 3 minutes each and further air dried at room temperature [12].

2.3 Preparation of ferrofluid

Ferrofluid is a fluid that becomes strongly magnetized in the presence of a magnetic field. In the absence of an external magnetic field, ferrofluid is unable to keep their magnetization (superparamagnetic). A small amount of the synthesized nanoparticles were dissolved in vegetable oil which resulted in the formation of a black slurry. In the presence of an external magnetic field (Neodymium magnet), the fluid jumps and forms spike like projections [13].

2.4 Determining the efficiency of iron oxide nanoparticles in the separation of oil spills from water

Three different petri plates were taken and labelled as Control, One drop of ferrofluid and Five drops of ferrofluid respectively. 14ml of water, a pinch of food color (for visibility) and 2.5 ml of vegetable oil was added to each petri plate. One drop of ferrofluid was added in the second petri plate and five drops of ferrofluid was added to the third petri plate. With the help of the magnet (the magnet was covered with a plastic to safeguard it), the oil was separated from water. The separation procedure was repeated for 2 more times for a total of three tests. After separation, the left over liquid in the petri plate was carefully transferred to a graduated cylinder without using a funnel. The average volume of leftover oil from the three tests was calculated and the result was recorded. The efficiency of separation by nanoparticles with the help of ferrofluid was calculated using the formula [13] :

$$1-\{\text{Volume of left over oil (ml)} / 2.5\text{ml}\}$$

III. RESULTS AND DISCUSSIONS

3.1 Visual Observation

The iron oxide nanoparticles were synthesized using *Andrographis paniculata* leaf extract. When the leaf extract, $FeCl_3$ and $FeCl_2$ solution was mixed, the color of the reaction changed instantaneously from light green to dark brown color. The blackish brown precipitates of iron oxide nanoparticles obtained after centrifugation of the above solution was further purified and characterized to know the surface morphology and the functional groups present. Characterization was done by UV-Visible Spectroscopy, XRD, FTIR, SEM and HR-TEM.

3.2 UV-Visible Spectroscopy Analysis

The formation of iron oxide nanoparticles was further studied by measuring the absorbance with UV-Visible Spectroscopy over the range from 200nm-700nm. The synthesized iron oxide nanoparticles exhibited an absorption peak within the range of 260nm-280nm, which a characteristic of it. These results are in harmony with the findings of Saranya et al., 2017 [14] who had reported green synthesis of iron oxide nanoparticles using aqueous extract of Musa ornata flower sheath.

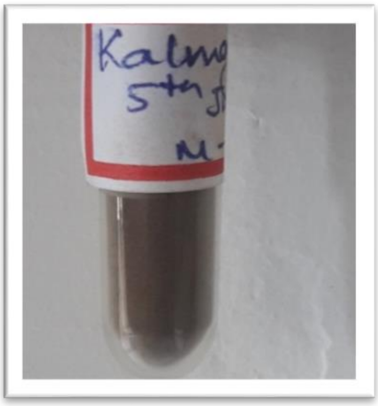


Fig 2: Powdered Iron Oxide Nanoparticles

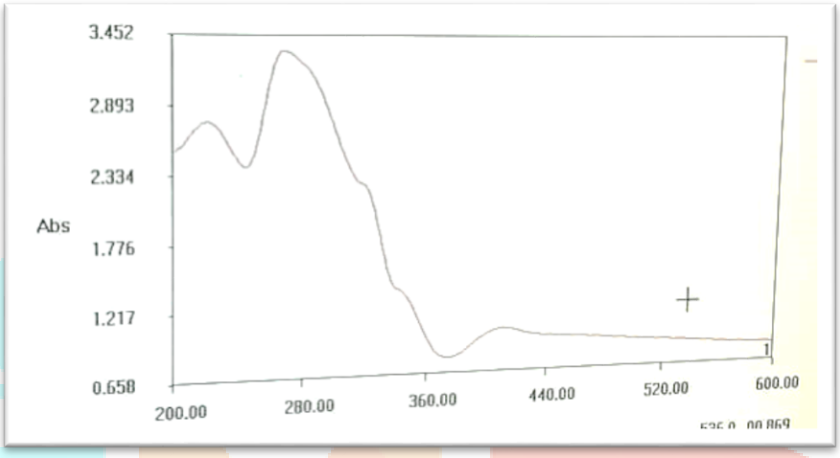


Fig 3: UV-Visible Spectrum of Synthesized Iron Oxide Nanoparticles

3.3 XRD Analysis

The powdered XRD pattern of the prepared iron oxide nanoparticles using A. paniculata leaf extract is shown in adjacent figure. The major strong characteristic peaks of iron oxide particles are obtained at 2θ =17.0˚ , 26.4˚, 28.26˚, 28.62˚, 34.90˚, 39.219˚, 45.371˚. The major characteristic peaks of iron oxide nanoparticles correspond to the crystalline structure of iron oxide nanoparticles. These findings are analogous with the crystalline nature of iron oxide nanoparticles Saranya et al.,2017 [14].

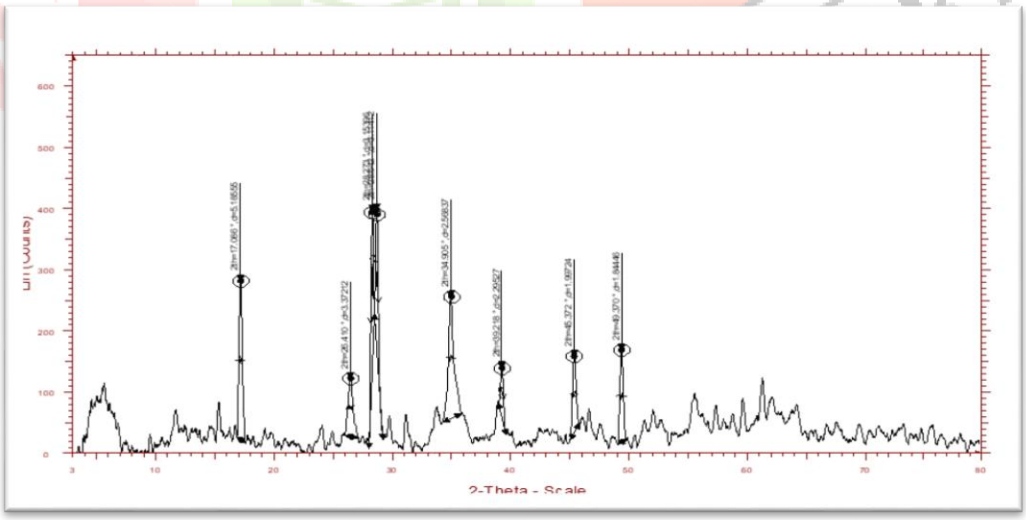


Fig 4: XRD Analysis of Synthesized Iron Oxide Nanoparticles

3.4 FT-IR Analysis

The FTIR analysis of synthesized iron oxide nanoparticles gave different stretching vibrations at different peaks , The presence of peak at 3384.62 cm−1 indicate the possible O-H stretching vibration of alcohol groups, 1630.84 cm−1 corresponds to C=C (alkene) stretching vibrations, 1415.15 cm−1 corresponds to C-H bending vibrations, 1384.6 cm−1 corresponds to N-O (nitro) stretching vibrations, 1205.04 cm−1 corresponds to C-N (amine) stretching vibrations, 1090.68 cm−1 corresponds to C-O (ether) stretching vibrations, 1016.36 cm−1 corresponds to C-F (alkyl halide) stretching vibrations, 846.89 cm−1, 630.98 cm−1 and 510.89 cm−1 corresponds to alkyl halide stretching vibrations of C-H, C-Cl, C-Br groups. The identified functional groups are found in previous FT–IR analysis of iron oxide nanoparticles synthesized by green tea extract by Gottimukkala et al,.2017 [15].

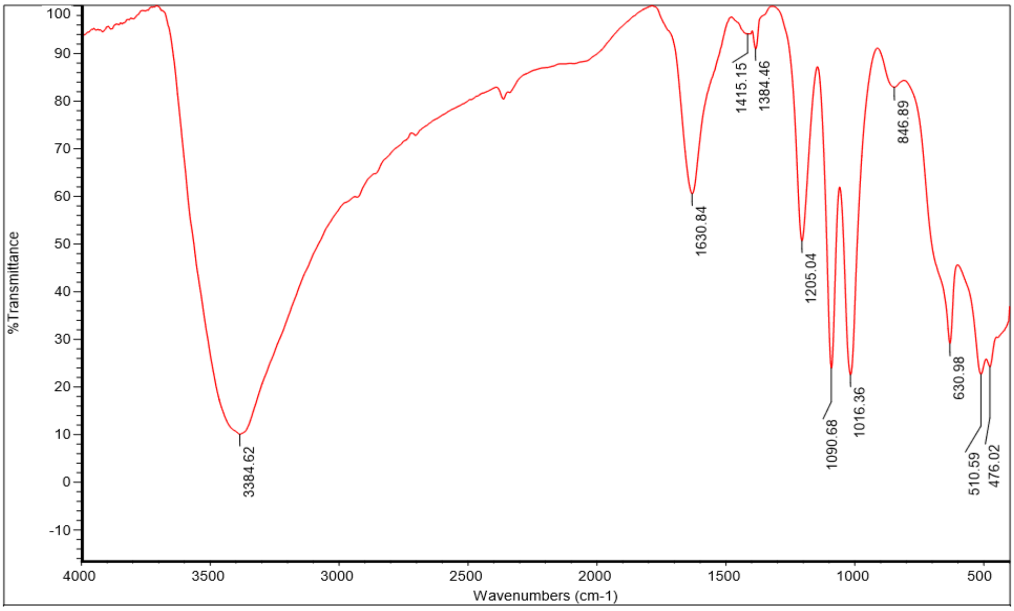


Fig 5: FT-IR Analysis of Synthesized Iron Oxide Nanoparticles

3.5 SEM Analysis

The morphological dimensions of synthesized iron oxide nanoparticles were studied using the SEM (scanning electron microscopy). The study demonstrated that the shape of the nanoparticle is irregular spherical, which are agglomerated together. Detailed structural information was further confirmed by HR- TEM. The result is in harmony with Kuang et al.,2013 [16] used three different tea extracts, namely, green tea, oolong tea and black tea to synthesis iron nanoparticles and the SEM image revealed the irregular spherical iron nanoparticles.

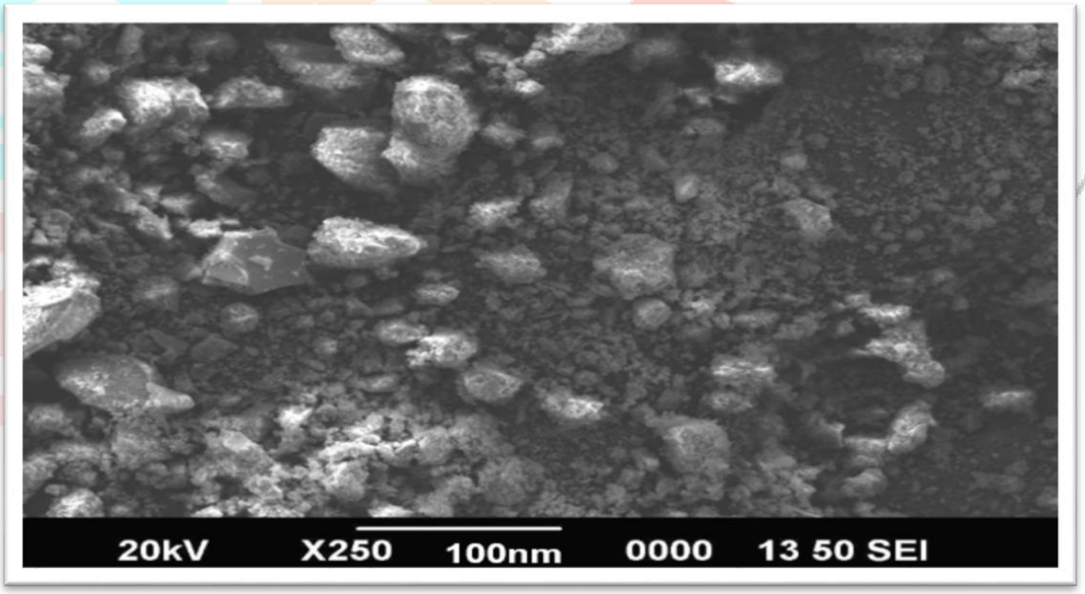


Fig 6: SEM Analysis of Synthesized Iron Oxide Nanoparticles

3.6 HR- TEM Analysis

Morphologies of the synthesized nanoparticles during the bio-reduction were confirmed by employing HR-TEM analysis. Iron oxide nanoparticles exhibited irregular spherical nanostructures with the average core diameter of 20nm. It is analogues to S. Sridhar et al., 2018 [12] who reported spherical iron oxide particles with a diameter of up to 20 nm were formed by *Glycosmis mauritiana*.

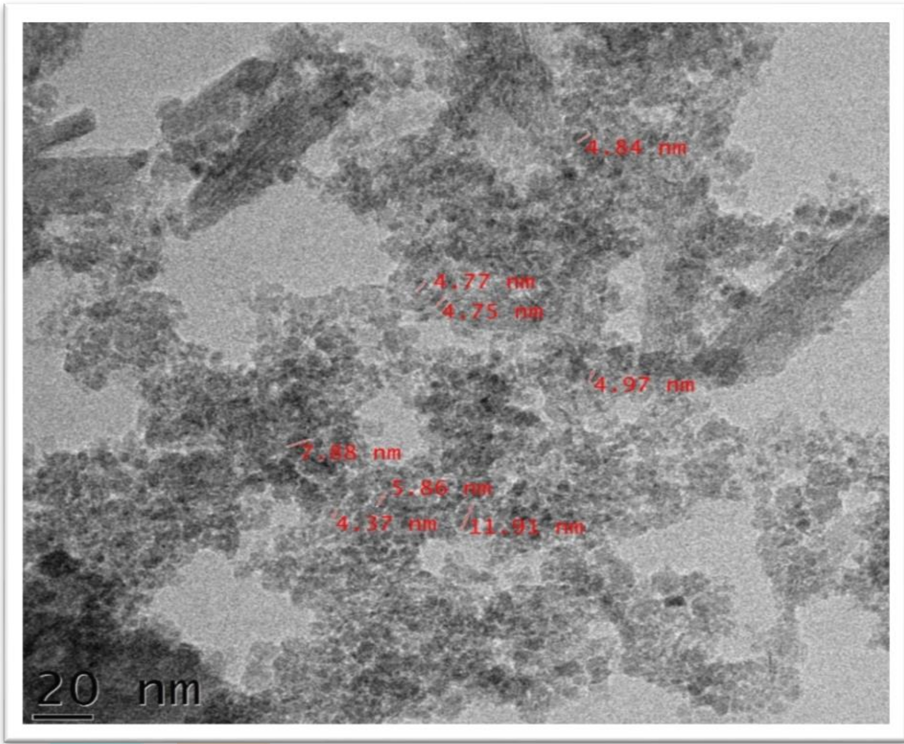


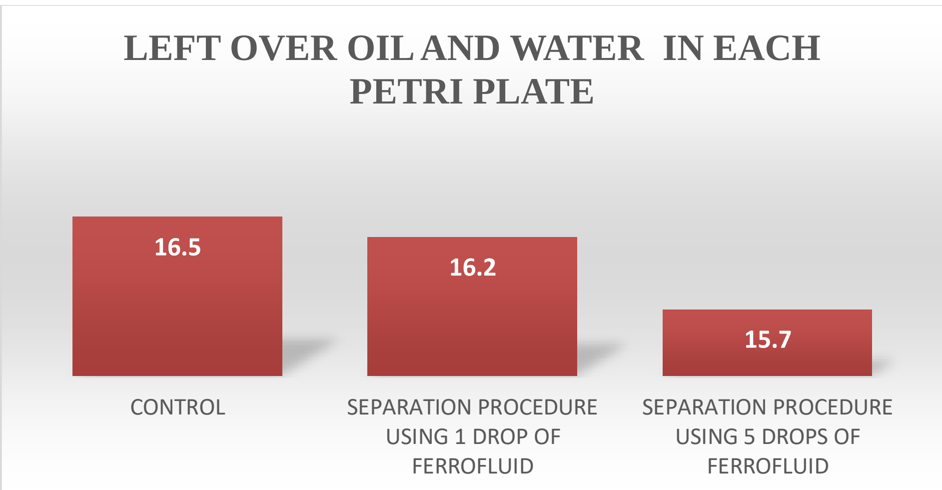
Fig 7: HR-TEM Analysis of Synthesized Iron Oxide Nanoparticles

3.7 Determining the efficiency of iron oxide nanoparticles in the separation of oil spills from water

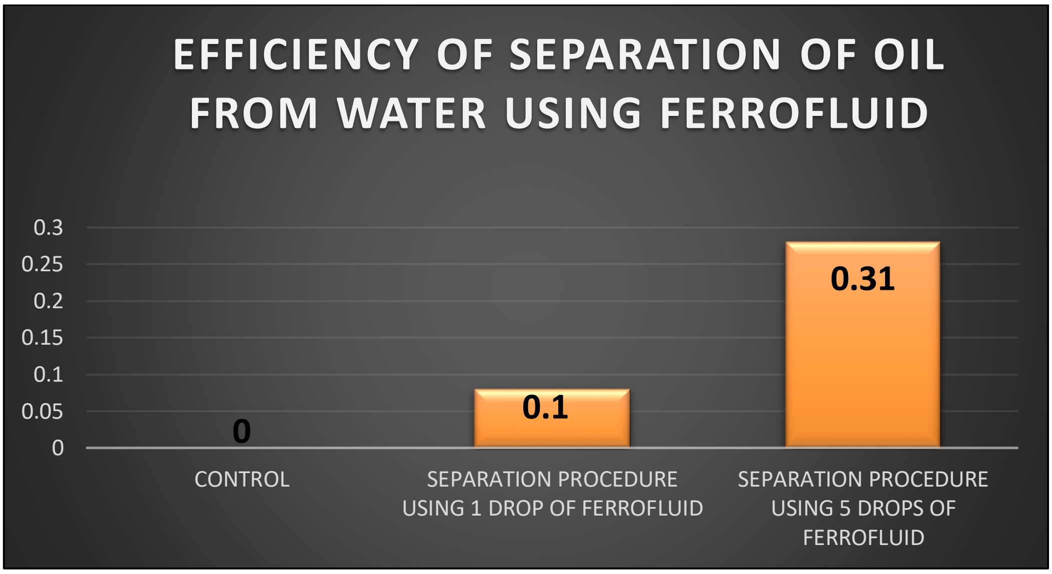
The magnetic property of the iron oxide nanoparticles was successfully exploited to be used for the formation of ferrofluid so as to separate the oil spill from water (on a small scale). The magnetic iron oxide nanoparticles in the ferrofluid gets dispersed into the and make the oil in the water magnetic, so in the presence of a strong magnetic field, the nanoparticle along with the oil present in the water gets separated.

Table 3.7: Observation Table for the left over oil and water in the petri plate

Sl. No	VOLUME OF LEFT OVER OIL AND WATER AFTER CLEANING PROCEDURE		
	CONTROL (ml)	CLEANING PROCEDURE USING 1 DROP OF FERROFLUID (ml)	CLEANING PROCEDURE USING 5 DROPS OF FERROFLUID (ml)
TEST 1	16.5	16.3	15.7
TEST 2	16.5	16.2	15.8
TEST 3	16.5	16.2	15.7
AVERAGE	16.5	16.2	15.7
VOLUME OF LEFT OVER OIL	2.5	2.2	1.7
EFFICIENCY	0	0.10	0.31



Graph 3.7.1: The above graph shows the volume of liquid left after the separation procedure



Graph 3.7.2: The above graph shows an increased efficiency in the separation of oil spill from water as the amount of ferrofluid is increased

The graph 3.7.1 and 3.7.2 shows an efficient separation of oil spill from water using ferrofluid. As the amount of ferrofluid in the water was increased, the separation procedure became more efficient.

IV. CONCLUSION

This is the first report on rapid and efficient green synthesis of iron oxide nanoparticle from the medicinal plant, Andrographis paniculata. The nanoparticles were almost spherical in shape with a core diameter of 20nm. The nanoparticles exhibited a crystalline structure which can in the future help in reducing the toxicity related to the nanoparticle in biomedical applications. The nanoparticle had many functional groups on its surface such as alkene, carbonyl group, etc., which can be further used to modify the nanoparticle according to the need of the study. It can be modified to achieve target drug delivery, can be used to make different formulations against various diseases and various other applications. The magnetic property of iron oxide nanoparticles was further used in the separation of oil spill from water (small scale). The nanoparticle was used to form the ferrofluid which jumps in the presence of an external magnetic field. The present research work revealed an increased efficiency in the separation of oil from water as the amount of ferrofluid was increased, thus aiding in reduction of one of the major reason for water pollution in oceans

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