Spin dynamic investigation and structural co-relation of the ensembles of two-dimensional primary nanosystem

UGC DAE CSR Project no. CRS/2021-22/0 1/594

Completion Report

2022-25

Name of the awardee: Dr. Pritam Deb
Professor,
Department of Physics
Tezpur University (Central University)
Tezpur-784028

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Achieved objectives:

- The following three systems of different morphologies and assembly structures have been developed, (a) α-Fe_2O_3 nanoflakes, (b) $FeNi_3$ nanobeads, (c) Cr_2O_3 nanosheets. The characterizations have been performed to determine their chemical properties.
- Quantitative morphological studies have been performed with the help of SANS and SAXS experiments which are then corroborated with the TEM, FESEM micrographs for all the systems.
- The d.c and a.c magnetic studies have been carried out to determine the intrinsic magnetic and interactions amongst the particles in a particular system.
- TD-NMR studies have been performed for the systems to gauge MR relaxivity properties.

Results and Discussion:

1.1 Development of α-Fe_2O_3 nanoflakes system:

A microwave assisted synthesis method has been employed to develop the 2D nanoflakes of α-Fe_2O_3. This procedure is based on the principle of liquid exfoliation method which depends upon liquid phase dispersion of the metal chloride in a suitable solvent. This dispersion is then subjected to microwave pulse shots which breaks the chloride-metal bonds and finally forms metal-oxide bonds under the pressurised reaction container at the appropriate temperature.

1.2 Morphological studies carried out via SAXS/SANS:

For the assessment of the type of morphology and interactions between them, SANS experiment was carried out for the system. The SANS intensity fitting has been performed over a logarithmic scale as shown in Fig.1 (a), using the scattering model for polydispersive interacting planar object with homogenous cross section. The form factor function for planar objects is given as,

$$P_{Planar}(Q) = P'(Q)\, P_{CS}(Q) \qquad (1)$$

where, the shape factor for the planar discs is given by

$$P'_{disc}(Q,R) = 2\pi^2 R^4/(QR)^2\{1 - (J_1(2QR))/QR\} \qquad (2)$$

and the shape factor for the cross section is given by

$$P_{CS}(Q,\eta,L) = \{\eta L \sin((QL)/2)\} / (QL)/2 \qquad (3)$$

There is a higher intensity that has been observed in the lower Q range which attributes to the presence of agglomeration in the system. Some of the fitted parameters that were obtained are the polydispersity index of 0.33, lateral width and the thickness of the flakes as 1.1 μm and 60 nm respectively. The interparticle distance between the nanoparticles has been found to be 400 nm. These results comply with that of the micrographs obtained from TEM or FESEM images for the sample, where the mean size of the flakes were observed to be about 900-1000 nm.

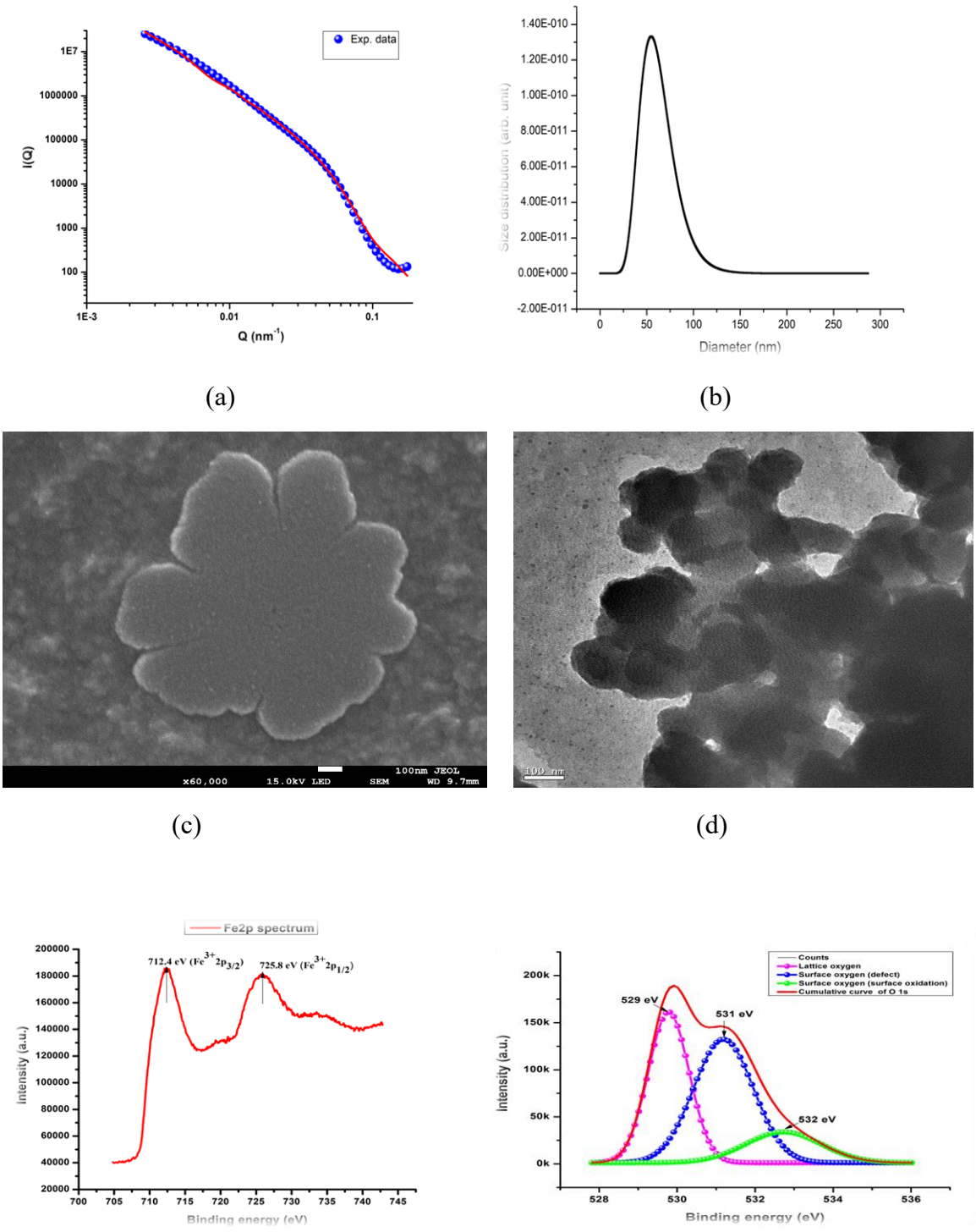
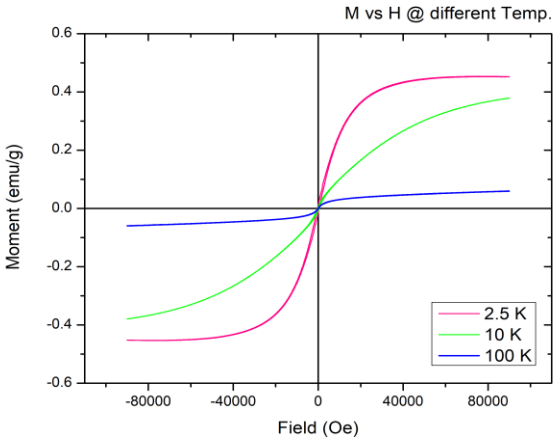


Fig.1 (a)SANS intensity fitting and (b) size distribution histogram; (c)FESEM and (d) TEM micrographs for α-Fe_2O_3 nanoflakes system. XPS spectra for (e)Fe 2p and (f)O 1s elements.

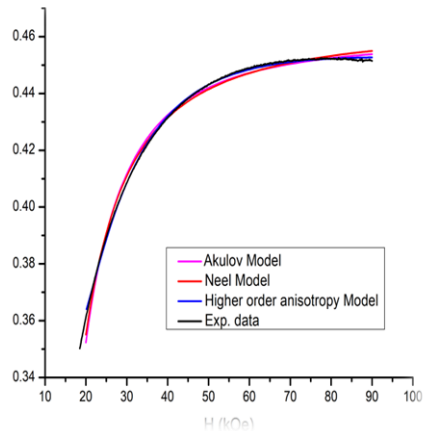
1.3 Magnetic measurements for α-Fe_2O_3 nanoflakes system:

The magnetic characterization of the as prepared 2D α-Fe_2O_3 nanoflakes were carried out in a 9T Vibrating Sample Magnetometer (VSM). Various thermo-magnetic characterizations were performed. The magnetization of the sample (M) in response to varying magnetic field (H) was studied for three different temperatures, viz., 2.5K, 10 K, and 100 K. The M/H plots are shown in Fig. 1 below. The M vs H plots for all three temperatures show a Hysteresis behaviour. From these graphs, different magnetic values have been calculated, like the saturation magnetization, the coercive field, etc. The magnetic saturation values decrease as well with increasing temperature, indicating a decline in the ferromagnetic distribution in the system at higher temperatures. From the hysteresis loops too, we observe a distinctive shape observed at 10 K as shown in Fig. 2. The linear nature of the curve seems to be dominating at this temperature indicating a magnetic arrangement steering away from ferromagnetic nature.

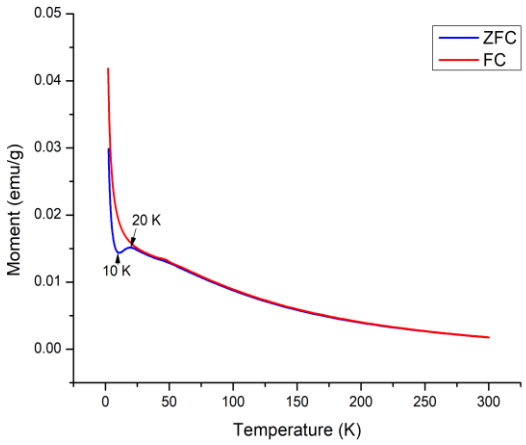
The thermo-magnetic study was carried out by plotting the Zero Field Cooled (ZFC) and Field Cooled (FC) curves at 100 Oe magnetic field. The temperature range varied from room temperature to almost 0K. Till 20 K, both the M_{ZFC} and M_{FC} curves stick together, monotonically increasing with lowering of the temperature. After this point, the two curves split before converging together again at very low temperatures. The split in the M_{ZFC} and M_{FC} curves usually occur when there is some type of magnetic frustration present inside the sample, like the coexistence of (antiferromagnetic) AFM and (ferromagnetic) FM phases. The M_{ZFC} curve forms a small peak around 20 K, where it could indicate the occurrence of superparamagnetic relaxation around that temperature in the sample.



(a)



(b)

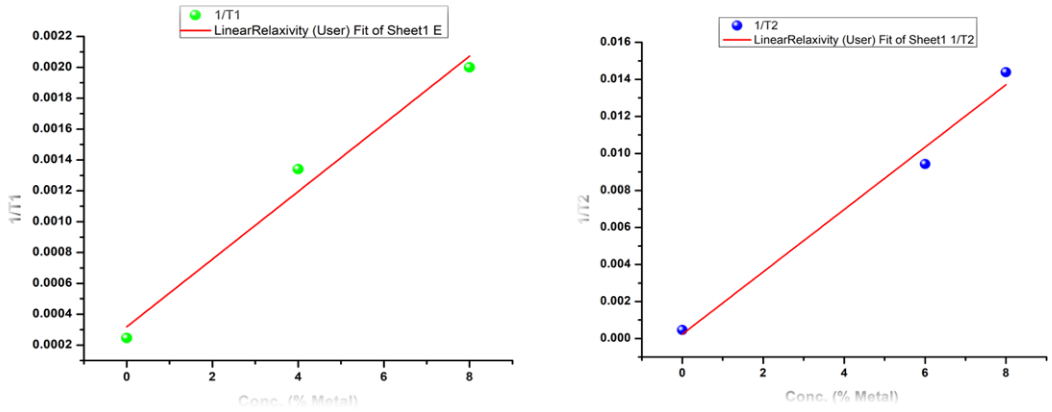


(c)

Fig.2 Thermo-magnetic studies for α-Fe_2O_3 nanoflakes, (a) M/H, (b) LAS fitting and (c) M/T graph

1.4 Magnetic relaxivity studies for α-Fe_2O_3 nanoflakes system:

MR relaxivity properties of any magnetic system gauges the type of decay mode to equilibrium it chooses when the magnetic atoms interact with the surrounding water molecules. This sheds light on the parameters of the longitudinal and transverse relaxation modes. This information is useful in calculating the two relaxation coefficients r_1 and r_2. The quantitative relationship amongst the two coefficients gives us an idea regarding the employability of the magnetic system as a possible T_1 or T_2 MRI contrast agents. For the current system, Time-domain NMR experiment was used on a series of nanomaterial solutions with increasing metal concentration with respect to the dispersing media, here chosen as DI water. As shown in Fig. 3, the linear fitting curves of the inverse of longitudinal and transverse times versus the metal concentration have been plotted. From the linear fit, the values of the slope, i.e., the relaxivity coefficients have been calculated to be 0.000219 $s^{-1}mM^{-1}$ and 0.00168 $s^{-1}mM^{-1}$ respectively. The ratio r_2/r_1 gives us a value of 7.9 which is less than 10. Hence, we can conclude from this study that the system of α-Fe_2O_3 nanoflakes can be employed as a dual mode MRI contrast agent.

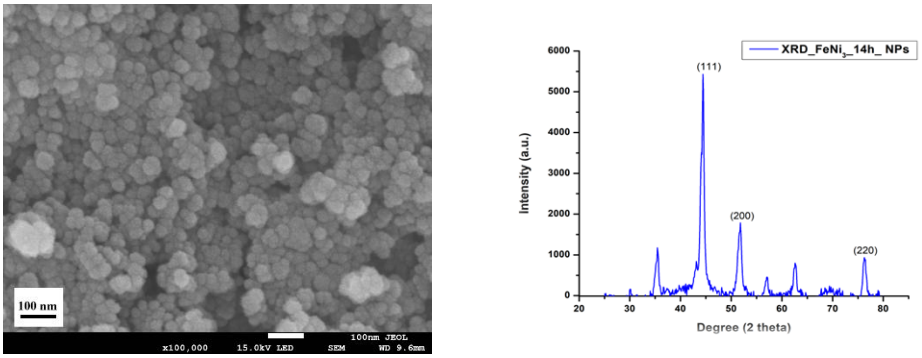


(a) (b)

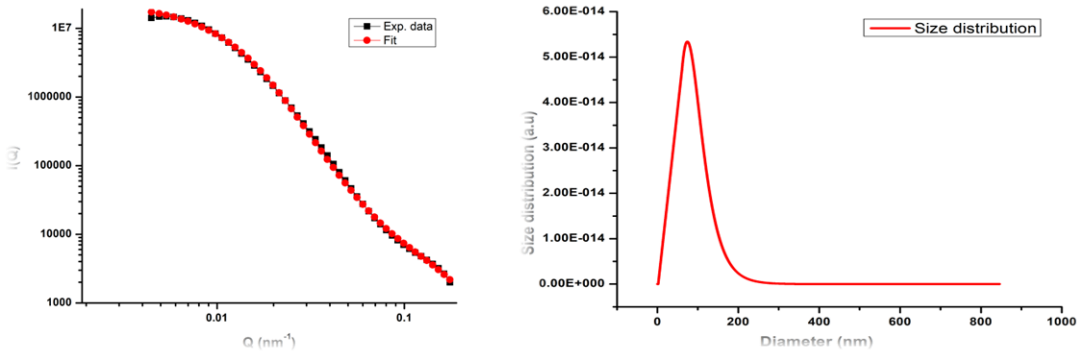
Fig. 3 TD-NMR study with metal concentration depedent variation of (a)longitudinal and (b) transverse relaxation times.

2.1 Development and magnetic properties of $FeNi_3$ system:

$FeNi_3$ nanobeads were synthesized via a solvothermal method, wherein the resultant product was found to be nanobeads of diameter varying between 25-35 nm as shown in Fig.4. The SANS intensity profile fitting was carried out which incorporated the spherical form factor. The polydispersity index was found to be 20% from the fitting parameters. For assessing the elemental analysis, XRD and EDX was done. The XRD intensity profile shows the three distinct peaks (111), (200), and (220) indexed at the angles 44^0, 51^0, and 75^0 degrees which are associated with the cubic phase of $FeNi_3$. The dc magnetic experiment shows a superparamagnetic nature of the system at a range of temperature from 3 K to 360 K. The thermos-magnetic study reveals the nature of temperature dependent properties, like the Blocking temperature of the system occurring at around 30 K.

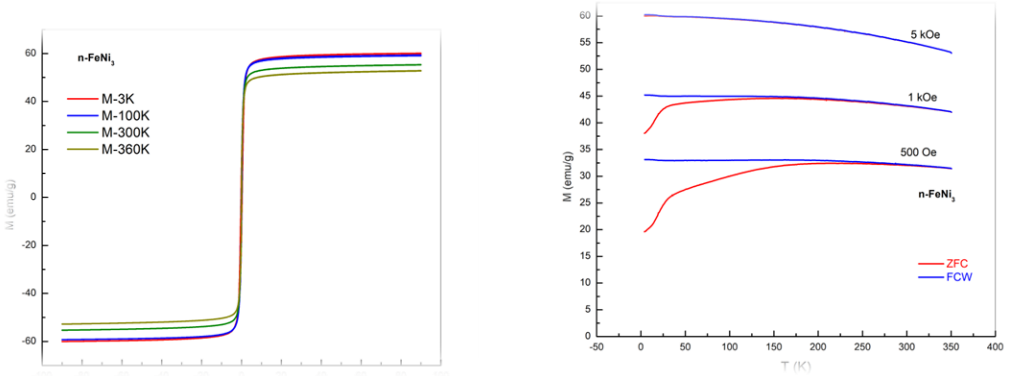


(a) (b)



(c) (d)

Fig.4. (a) FESEM micrograph, (b) XRD spectrum, (c) SANS intensity fitting and (d) size distribution curves for $FeNi_3$ nanobeads.



(a) (b)

Fig.5. Thermo-magnetic studies for $FeNi_3$ nanobeads, (a) M/H and (b) M/T curves.

3.1 Development of Cr_2O_3 nanosheets via microwave-assisted method:

2D nanosheets of size 300 nm in length and a few nanometres in width have been developed with the help of a microwave -assisted method. The intrinsic magnetic properties of the system have been studied with the help of the VSM instrument sweeping across the temperature range from 2 to 300 K. The liquid sample was dried onto a silica substrate, which was then loaded onto the glass boat to be loaded into the VSM chamber. The M/H response shows a superparamagnetic nature at 2 K, which shows that at such low temperatures the magnetic dipolar interactions are much stronger than the thermal energy contributions. To verify the superparamagnetic nature of the system, the Langevin's fit was employed over the M/H data which corroborated with the value of the magnetic parameters such as the magnetic saturation value as well.

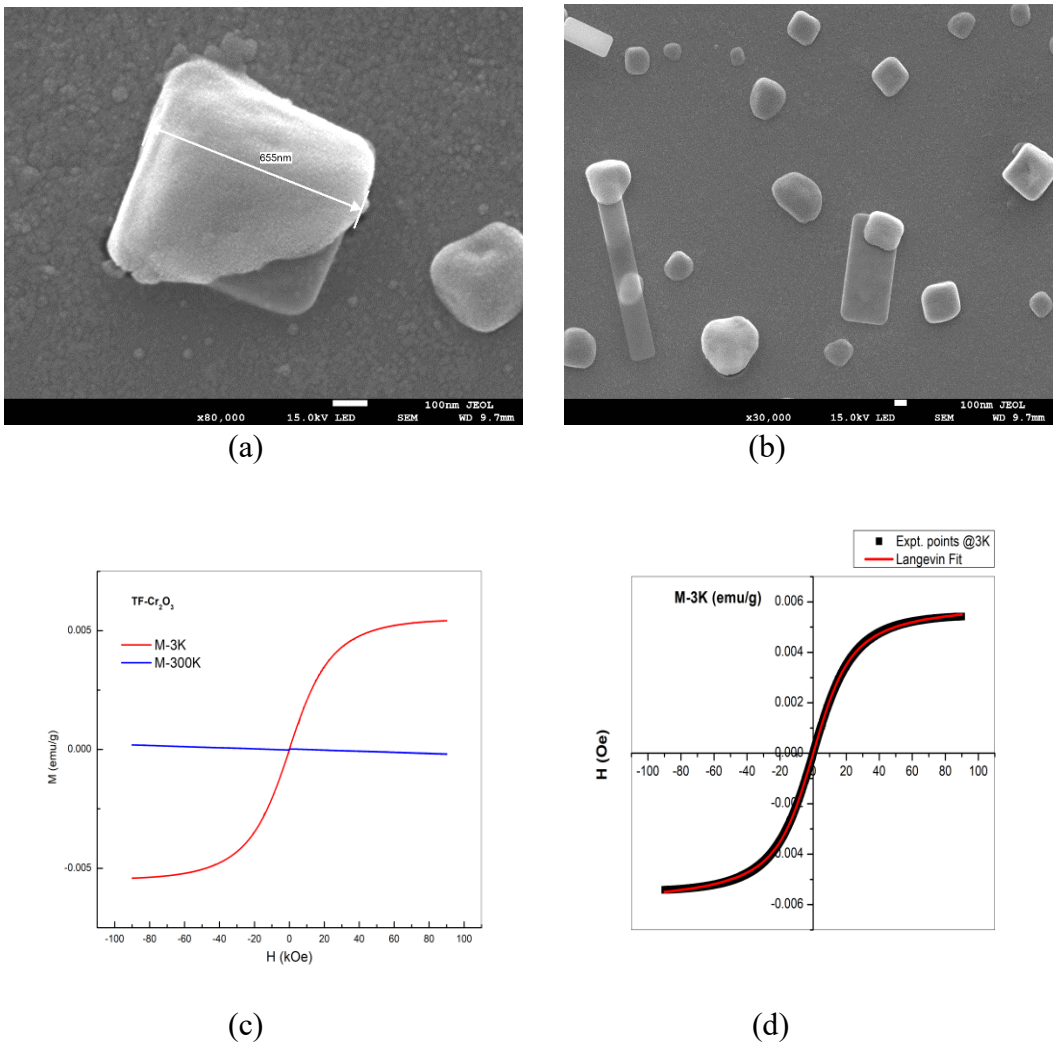


Fig.6 (a)FESEM, (b) TEM micrographs, (c) and (d) M/H curves and LAS fitting

New leads obtained:

We have developed a unique morphology of the α-Fe_2O_3 nanoparticles. Flower-shaped flat nanoflakes have been observed from micrographs. The intrinsic magnetic properties have been studied which show a superparamagnetic nature of the system across a wide temperature range. The developed nanosystem also shows promising applicability as a dual mode MR contrast agent from both the TD-NMR studies and the phantom images obtained from MRI experiments. The ratio r_2/r_1 giving a value between 3 and 10 shows that the nanosystem can be utilized a dual mode contrast agent for various biomedical applications.

The developed systems of $FeNi_3$ nanobeads and the 2D Cr_2O_3 nanosheets also promising magnetic characteristics at a wide range of temperatures. The applications as possible contrast agents of these two systems are currently in progress.

Publications from this project:

Korobi Konwar, Som Datta Kaushik, Peram Delli Babu, Anamika Chaturvedi, Dinesh Kumar, Rituraj Chakraborty, Rupak Mukhopadhyay, Pooja Sharma, Saurabh Lodha, Debasis Sen and Pritam Deb; **Integrative Modulation of Magnetic Resonance Transverse and Longitudinal Relaxivity in a Cell-Viable Bimagnetic Ensemble, γ-Fe2O3@ZnFe2O4**; *Langmuir 2024*, 40, 3, 1793–1803

References:

1) Korobi Konwar, Niyorjyoti Sharma, Pranjali Pranjali, Anupam Guleria, Som Datta Kaushik, Anupam Dutta, Rupak Mukhopadhyay, Debasis Sen, Weibo Gao, and Pritam Deb, Langmuir 2022 38 (36), 11087-11098
2) Korobi Konwar, Mayuri Bora, Som Datta Kaushik, Anamika Chaturvedi, Dinesh Kumar, Anupam Dutta, Rupak Mukhopadhyay, Peram Delli Babu, Pooja Sharma, Saurabh Lodha, Debasis Sen, Pulickel M. Ajayan, and Pritam Deb, ACS Applied Nano Materials 2023 6 (21), 20440-20457
3) Zhou, Z., Yang, L., Gao, J., Chen, X., *Adv. Mater.* 2019, 31, 1804567
4) S.-h. Noh, W. Na, J.-t. Jang, J.-H. Lee, E. J. Lee, S. H. Moon, Y. Lim, J.-S. Shin, J. Cheon, Nano Lett. 2012, 12, 3716
5) You-Cun Chen, Fang-Cai Zheng, Yu-Lin Min, Tao Wang, Ying-Guo Zhao, Solid State Sciences, 2012, ISSN 1293-2558
6) M. E. Schabes, H. N. Bertram, J. Appl. Phys. 1988, 64, 1347
7) J. Mater. Chem. B, 2017,5, 3629-3633
8) Nanoscale, 2014,6, 13637-13645
9) Anal. Chem. 2021, 93, 14, 5691–5699
10) Nano Lett. 2012, 12, 7, 3716–3721
11) ACS Materials Lett. 2021, 3, 5, 631–640
12) K. Saikia et al., Applied Surface Science, Volume 464, 2019,

UTILISATION CERTIFICATE

Certified That out of Rs. Two lakhs twenty eight thousand one hundred twenty (Rs. 2,28120.00) of grants-in-aid sanctioned during the year 2022-23 in favour of Prof. Pritam Deb under UGC-DAE Consotrium of Scientific Research letter/Order No. 2022-23/2274 dated 31.03.2022 and Rs. NA on account of unspent balance of the previous year, a sum of Rs. One lakh forty six thousand one hundred eleven (Rs. 146,111.00) has been utilised for the purpose of execution of the project for which it was sanctioned and that the balance of Rs. Eighty two thousand and nine (82,009.00) remainig unutilised at the end of the year has been surrendered to the UGC-DAE Consotrium of Scientific Research (CRS/2021-22/01/594) will be adjusted towards the grants-in-aid payable during the next year i.e. 2023-24

(Principal Investigator)

Dr. P. DEB
Professor
Dept. of Physics
TEZPUR UNIVERSITY
(Central University)

Date

Finance Officer

19.4.23

Date :

Finance Officer
Tezpur University

Signature of the Head of the Institute

Date :

Registrar
Tezpur University

(To be filled by UGC-DAE CSR)

Certified that I have satisfied that the conditions on which the grants-in-aid ws sanctioned have been fulfilled/are being fuilfilled and that I have excercised the following checks to see that the money was actually utililsed for the purpose for which it

Kinds of Checks exercised.

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Signature :_____

Designation:_____

Date :_____

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR 2022-23

Annexure-III

1	Title of the Project/Scheme :		**Spin dynamics investigation and structural co-relation of the ensembles of two-dimensional primary nanosystem**	
2	Name of the Institution		**Tezpur University**	
3	Principal Investigator		**Prof. Pritam Deb**	
4	UGC-DAE Consortium for Scientific Research sanction order No & date sanctioning the project		**CRS/2021-22/01/594, 24.08.2022**	
5	Head of account as given in the original sanction order		**Prof. Pritam Deb**	
6	Amount brought forward from the previous financial year quoting UGC-DAE Consortium for Scientific Research letter no & date in which th authority to carry forward the said amount was given	**i.** Amount (Rs.)		**NA**
		ii. Letter No.		
		iii. Date		
7	Amount received during the financial year	**i.** Amount (Rs.)		**228,120.00**
		ii. Letter/Order No		**2022-23/2274**
		iii. Date		**31.03.2022**
8	Total amount that was available for expenditure (excluding commitments) during the financial year (Sl No 6+7)	**Rs.**	**228,120.00**	
9	Actual expenditure (Excluding commitments)	**Rs.**	**146,111.00**	
10	Balance amount available at the end of the financial year	**Rs.**	**82,009.00**	
11	Unspent balance refunded, if any	**Rs.**	-	
12	Amount to be carried forward to the next financial year	**Rs.**	**82,009.00**	

(Principal Investigator)
Dr. P. DEB
Professor
Dept. of Physics
TEZPUR UNIVERSITY
(Central University)

Finance Officer
Finance Officer
Tezpur University

20/4/23
Signature of the Head of the Institute
Registrar
Tezpur University

Statement of Expenditure
for the Financial Year 2022-23

Sl No	Sanctioned Heads	Total Funds Allocated (Sanctioned)	Expenditure Incurred			Total Expenditure till _____	Balance as on ___	Remarks (If any)
			1st Year	*2nd Year*	*3rd Year*			
(I)	(II)	(III)	(IV)	(V)	(VI)	(VII=IV+V+VI)	(VIII=III-VII)	
	Student Fellowship	183,120.00	101,161.00			101,161.00	81,959.00	
	Consumables	30,000.00	29,979.00			29,979.00	21.00	
	Contingency	15,000.00	14,971.00			14,971.00	29.00	
						-	-	
						-	-	
						-	-	
						-	-	
8	**Total**	**228,120.00**	**146,111.00**	-	-	**146,111.00**	**82,009.00**	

(Principal Investigator)

Dr. P. DEB
Professor
Dept. of Physics
TEZPUR UNIVERSITY
(Central University)

Date :

Finance Officer
19.4.23

Finance Officer
Tezpur University

Date :

Signature of the Head of the Institute

20/4/23

Registrar
Tezpur University

UTILISATION CERTIFICATE

Certified That out of Rs. **One lakh forty six thousand one hundred fifty six (Rs. 146,156)** of grants-in-aid sanctioned during the year 2023-24 in favour of **Prof. Pritam Deb** under UGC-DAE Consotrium of Scientific Research letter/Order No. **CRS/2021-22/03/594** dated **15.05.2023** and Rs. **Eighty two thousand and nine(Rs. 82,009)** on account of unspent balance of the previous year, a sum of **Rs. Two lakhs thirteen thousand and one hundred fifty one(Rs. 213,151)** has been utilised for the purpose of execution of the project for which it was sanctioned and that the balance of **Rs. Fifteen thousand and fourteen(Rs. 15,014)** remaining unutilised at the end of the year has been surrendered to the UGC-DAE Consotrium of Scientific Research **(CRS/2021-22/01/594)** will be adjusted towards the grants-in-aid payable during the next year i.e. **2024-25**

(Principal Investigator)

Dr. P. Deb
Professor
Dept. of Physics
Tezpur University, Tezpur- 784028

Date

Finance Officer

Finance Officer
Tezpur University

Date : 14.5.24

Signature of the Head of the Institute

Registrar
Tezpur University

Date :

(To be filled by UGC-DAE CSR)

Certified that I have satisfied that the conditions on which the grants-in-aid ws sanctioned have been fulfilled/are being fuilfilled and that I have excercised the following checks to see that the money was actually utililsed for the purpose for which it

Kinds of Checks exercised.

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Signature :_____

Designation:_____

Date :_____

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR 2023-24

Annexure-III

1	Title of the Project/Scheme :		**Spin dynamics investigation and structural co-relation of the ensembles of two-dimensional primary nanosystem**	
2	Name of the Institution		**Tezpur University**	
3	Principal Investigator		**Prof. Pritam Deb**	
4	UGC-DAE Consortium for Scientific Research sanction order No & date sanctioning the project		**CRS/2021-22/01/594, 24.08.2022**	
5	Head of account as given in the original sanction order		**Prof. Pritam Deb**	
6	Amount brought forward from the previous financial year quoting UGC-DAE Consortium for Scientific Research letter no & date in which th authority to carry forward the said amount was given	**i.** Amount (Rs.)	**82,009.00**	
		ii. Letter No.	**CRS/2021-22/03/594**	
		iii. Date	**15.06.2023**	
7	Amount received during the financial year	**i.** Amount (Rs.)	**146,156.00**	
		ii. Letter/Order No	**CRS/2021-22/03/594**	
		iii. Date	**15.05.2023**	
8	Total amount that was available for expenditure (excluding commitments) during the financial year (Sl No 6+7)	**Rs.**	**228,165.00**	
9	Actual expenditure (Excluding commitments)	**Rs.**	**213,151.00**	
10	Balance amount available at the end of the financial year	**Rs.**	**15,014.00**	
11	Unspent balance refunded, if any	**Rs.**	-	
12	Amount to be carried forward to the next financial year	**Rs.**	**15,014.00**	

(Principal Investigator)
Dr. P. Deb
Professor
Dept. of Physics
Tezpur University, Tezpur- 784028

14.5.24
Finance Officer
Finance Officer
Tezpur University

Signature of the
Head of the Institute
Registrar
Tezpur University

Statement of Expenditure
for the Financial Year 2023-24

Sl No	Sanctioned Heads	Total Funds Allocated (Sanctioned) (First year + Second year)	Expenditure Incurred			Total Expenditure till 31.03.2024	Balance as on ___	Remarks (If any)
			1st Year	*2nd Year*	*3rd Year*			
(I)	(II)	(III)	(IV)	(V)	(VI)	(VII=IV+V+VI)	(VIII=III-VII)	
	Student Fellowship	284,276.00	101,161.00	168,000.00		269,161.00	15,115.00	
	Consumables	60,000.00	29,979.00	29,945.00		59,924.00	76.00	
	Contingency	30,000.00	14,971.00	15,206.00		30,177.00	(177.00)	
						-	-	
						-	-	
						-	-	
						-	-	
8	**Total**	**374,276.00**	**146,111.00**	**213,151.00**	**-**	**359,262.00**	**15,014.00**	

(Principal Investigator)

Date : Dr. P. Deb
Professor
Dept. of Physics
Tezpur University, Tezpur- 784028

Date :

14.5.24
Finance Officer
Finance Officer
Tezpur University

Signature of the Head of the Institute Registrar

Date : 14/5/24 Tezpur University

UTILISATION CERTIFICATE

Certified That out of **Rs. Two lakhs thirteen thousand one hundred six (Rs. 213,106)** of grants-in-aid sanctioned during the year 2024-25 in favour of **Prof. Pritam Deb** under UGC-DAE Consotrium of Scientific Research letter/Order No. **CRS/2021-22/03/594** dated **05.02.2025** and **Rs. Fifteen thousand and thirty two (Rs. 15,014)** on account of unspent balance of the previous year, a sum of **Rs. Two lakhs thirteen thousand and fifty eight(Rs. 212,981)** has been utilised for the purpose of execution of the project for which it was sanctioned and that the balance of **Rs. Fifteen thousand one hundred and thirty nine(Rs. 15,139)** remaining unutilised at the end of the year has been surrendered to the UGC-DAE Consotrium of Scientific Research **(CRS/2021-22/01/594)** will be adjusted towards the grants-in-aid payable during the next year i.e. **2025-26**

(Principal Investigator)

Date Dr. P. Deb Professor Dept. of Physics Tezpur University, Tezpur 784028

Finance Officer

Date : 22/6/25 Finance Officer Tezpur University

Signature of the Head of the Institute

Date : 27/06/2025 Registrar Tezpur University

(To be filled by UGC-DAE CSR)

Certified that I have satisfied that the conditions on which the grants-in-aid ws sanctioned have been fulfilled/are being fuilfilled and that I have excercised the following checks to see that the money was actually utililsed for the purpose for which it

Kinds of Checks exercised.

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2
3
4
5

Signature :_____

Designation:_____

Date :_____

Annexure-III

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR 2024-25

1	Title of the Project/Scheme :		Spin dynamics investigation and structural co-relation of the ensembles of two-dimensional primary nanosystem
2	Name of the Institution		Tezpur University
3	Principal Investigator		Prof. Pritam Deb
4	UGC-DAE Consortium for Scientific Research sanction order No & date sanctioning the project		CRS/2021-22/01/594, 24.08.2022
5	Head of account as given in the original sanction order		Prof. Pritam Deb
6	Amount brought forward from the previous financial year quoting UGC-DAE Consortium for Scientific Research letter no & date in which th authority to carry forward the said amount was given	i. Amount (Rs.) ii. Letter No. iii. Date	15,014.00 CRS/2021-22/03/594 05.02.2025
7	Amount received during the financial year	i. Amount (Rs.) ii. Letter/Order No iii. Date	2,13,106.00 CRS/2021-22/03/594 05.02.2025
8	Total amount that was available for expenditure (excluding commitments) during the financial year (Sl No 6+7)	Rs.	2,28,120.00
9	Actual expenditure (Excluding commitments)	Rs.	2,12,981.00
10	Balance amount available at the end of the financial year	Rs.	15,139.00
11	Unspent balance refunded, if any	Rs.	-
12	Amount to be carried forward to the next financial year	Rs.	15,139.00

(Principal Investigator)

Finance Officer
Finance Officer
Tezpur University

02/07/2025
Signature of the
Head of the Institute
Registrar
Tezpur University

Statement of Expenditure
for the Financial Year 2024-25

Sl No	Sanctioned Heads	Total Funds Allocated (Sanctioned) (First year + Second year+Third year)	Expenditure Incurred			Total Expenditure till 31.03.2024	Balance as on ___	Remarks (If any)
			1st Year	*2nd Year*	*3rd Year*			
(I)	(II)	(III)	(IV)	(V)	(VI)	(VII=IV+V+VI)	(VIII=III-VII)	
	Student Fellowship	4,52,382.00	1,01,161.00	1,68,000.00	1,68,000.00	4,37,161.00	15,221.00	
	Consumables	90,000.00	29,979.00	29,945.00	29,999.00	89,923.00	77.00	
	Contingency	45,000.00	14,971.00	15,206.00	14,982.00	45,159.00	(159.00)	
						-	-	
						-	-	
						-	-	
						-	-	
8	Total	5,87,382.00	1,46,111.00	2,13,151.00	2,12,981.00	5,72,243.00	15,139.00	

(Principal Investigator)

Date :

Dr. P. Deb
Professor
of Physics
Tezpur- 784028

Finance Officer

Date : 30/6/25

Finance Officer
Tezpur University

Signature of the Head of the Institute

02/07/2025

Date :

Registrar
Tezpur University

Progress Report

CRS proposal Number: CRS/2021-22/03/594

Name of the PI: Prof. Pritam Deb

PI's Institute: Tezpur University

Title of the Proposal: Spin dynamic investigation and structural co-relation of the ensembles of two dimensional primary nanosystem

Name of the Principal Collaborator (PC): Dr. S. D. Kaushik

Name of Centre at which the PC is associated: Mumbai

Objectives as stated in the approved proposal:

- Understanding of type of interaction among the ensemble of two-dimensional magnetic nanosystem
- Investigation on structural correlation using small angle X-ray Scattering and Small Angle Neutron Scattering, powder neutron diffractometer for crystal and magnetic structure aspect.
- The dependency of interaction on the dynamic magnetic responses by considering the direct-current susceptibility study
- The understanding of combined effect in proximity effect in the interface and demagnetizing effect among the primary nanoflakes and their behaviour on spin relaxation study.
- The in-depth phase transition investigation using AC susceptibility, Polarized Neutron Reflectometry (PNR).

Summary of the work done (within 250 words) and details of journal publications including the names of all authors:

Along with the shape or size of the MNPs, the dimension of the system may also play a factor on their magnetic parameters. The correlation between the lateral confinement of spins and the corresponding manifestation of the magnetic characteristics has not been delved into deeply yet. Thus, with this motivation we have chosen an iron oxide system. A two-dimensional structure of iron oxide (Hematene) has been synthesized using a facile synthesis process. Raman spectra of both the experimental samples show the same signature peaks at around 587, 800 and 1100 cm^{-1}. The peaks around 500 and 800 may be correlated to the T2g and A2g peaks found in the Raman spectra of gamma phase of iron oxide. When compared to that of the reference HRTEM images of 2D iron oxide, the prepared sample shows clustered flakes with a higher degree of opacity. This indicates that the thickness of the prepared sample is more than that what is desired for thin sheets of the reduced bulk iron oxide. To get the magnetization profile of the prepared samples, d.c. magnetization studies have been done. The Magnetization (M) vs Field (H), and M vs Temperature (T) profiles are being currently studied and analyzed. The correlation between the reduced dimension of the alpha-iron oxide nanoflakes and the corresponding magnetic properties at different temperatures will be subsequently established after the analysis of the procured data from the latest visit.

Journal publications under this project:

[1] Korobi Konwar, Som Datta Kaushik, Peram Delli Babu, Anamika Chaturvedi, Dinesh Kumar, Rituraj Chakraborty, Rupak Mukhopadhyay, Pooja Sharma, Saurabh Lodha, Debasis Sen and Pritam Deb; *Integrative Modulation of Magnetic Resonance Transverse and Longitudinal Relaxivity in a Cell-Viable Bimagnetic Ensemble, γ-Fe_2O_3@$ZnFe_2O_4$*; *Langmuir* 2024, 40, 3, 1793–1803
Publication date:
January 5, 2024

Details of the visits to UGC-DAE CSR:

Name of the visitor (student/PI)	Facilities used	Date and duration
Sayoree Purakayastha/Prof. Pritam Deb	M/H, M/T studies (PPMS), SANS-II	12/02/2024 – 16/02/2024 (4 days)

Difficulties in availing the facilities of UGC-DAE CSR:

Statement of accounts:

Fund received		Expenditure made as on date (19/02/2024)		Available balance	
Contingency	Fellowship	Contingency	Fellowship	Contingency	Fellowship
15,000/-	183,115/-	11,546/-	126,000/-	3,454/-	57,115/-

Pritam Deb.

Signature of the PI

Progress Report

CRS proposal Number: CRS/2021-22/03/594

Name of the PI: Prof. Pritam Deb

PI's Institute: Tezpur University

Title of the Proposal: Spin dynamic investigation and structural co-relation of the ensembles of two dimensional primary nanosystem

Name of the Principal Collaborator (PC): Dr. S. D. Kaushik

Name of Centre at which the PC is associated: Mumbai

Objectives as stated in the approved proposal:

- Understanding of type of interaction among the ensemble of two-dimensional magnetic nanosystem
- Investigation on structural correlation using small angle X-ray Scattering and Small Angle Neutron Scattering, powder neutron diffractometer for crystal and magnetic structure aspect.
- The dependency of interaction on the dynamic magnetic responses by considering the direct-current susceptibility study
- The understanding of combined effect in proximity effect in the interface and demagnetizing effect among the primary nanoflakes and their behaviour on spin relaxation study.
- The in-depth phase transition investigation using AC susceptibility, Polarized Neutron Reflectometry (PNR).

Summary of the work done (within 250 words) and details of journal publications including the names of all authors:

The magnetic study of the previously prepared nano system of 2D Iron oxide nanoflakes was concluded with the d.c. magnetic data. The superparamagnetic state of the system was established across all temperature ranges, with the Blocking temperature observed at around 20 K. The morphology of the 2D IONFs was quantitatively analyzed with the SANS data, using the log normal distribution for planar discs with homogenous cross section. Other morphology related parameters like the polydispersity index, lateral width, and thickness of the nanoflakes were also established with the help of the SANS fitting. The magnetic relaxation properties were studied with the help of TD-NMR and MRI experiments conducted at 1.41 and 3T respectively. The concentration dependent increase in both the longitudinal and transverse relaxation coefficients was observed. Thus, the applicability of the 2D IONFs as a potential MRI contrast agent was thus established with this study.
The magnetic and SANS measurements were also completed for the system of $FeNi_3$ nanobeads. The system is showing a superparamagnetic state, with a saturation value of about 60 emu/g at room temperature. The magnetization versus temperature data taken at different field strengths showed a hump like peak related to the blocking state found in superparamagnetic state at about 50 K. The a.c. magnetic data is yet to be completed for the complete magnetic analysis of this system.

Details of the visits to UGC-DAE CSR:

Name of the visitor (student/PI)	Facilities used	Date and duration
Sayoree Purakayastha/Prof. Pritam Deb	M/H, M/T studies (PPMS), SANS-II	23/12/2024 – 27/12/2024 (4 days)

Difficulties in availing the facilities of UGC-DAE CSR:

Statement of accounts:

Fund received		Expenditure made as on date (19/02/2024)		Available balance	
Contingency	Fellowship	Contingency	Fellowship	Contingency	Fellowship
15,000/-	168,106/-	15,000/-	168,000/-	NA	106/-

Signature of the PI

(1) Title of the project: Spin dynamic investigation and structural co-relation of the ensembles of two dimensional primary nanosystem

(2) Name and affiliation of the PI: Prof. Pritam Deb, Tezpur University

(3) Name of CSR Scientist or PC: Dr. Som Datta Kaushik, UGC-DAE CSR Mumbai Centre

(4) Approved objectives:

- Understanding of type of interaction among the ensemble of two-dimensional magnetic nanosystem
- Investigation on structural correlation using small angle X-ray Scattering and Small Angle Neutron Scattering, powder neutron diffractometer for crystal and magnetic structure aspect.
- The dependency of interaction on the dynamic magnetic responses by considering the direct-current susceptibility study
- The understanding of combined effect in proximity effect in the interface and demagnetizing effect among the primary nanoflakes and their behaviour on spin relaxation study.
- The in-depth phase transition investigation using AC susceptibility, Polarized Neutron Reflectometry (PNR).

(5) Objectives achieved so far:

Preliminary samples of nanosystems have been synthesized till now, and the magnetic characterizations and SANS/SAXS studies are being carried out at present.

(6) Details of work done with a few figures (not more than 2 pages):

Intrinsic magnetism in 2D nanosystems is still an undelved area at large presently. The first experimental evidence of 2D magnetism found in $Cr_2Ge_2Te_6$, magnetic ordering was found in the bilayers, though the ordering was lost in the monolayer limit. Other than the van der Waals systems, the recent upsurge in the two dimensional non van der Waals materials has steered the focus on exploring the magnetic properties in the same. 2D iron oxide was synthesized using a microwave-assisted liquid phase exfoliation (LPE) method. The synthesis procedure was carried out by using ferric chloride as the precursor and dimethyl formamide (DMF). The sealed glass tube used inside the microwave acts as a pressurized vessel where the metal chloride being a good dielectric, absorbs the electromagnetic waves generated inside the microwave cavity.

In order to verify the final product formed, i.e., iron oxide, FTIR of the sample was carried out. The FTIR of the sample is shown in Fig. 1 and was compared with reference FTIR responses of bulk iron oxide and solvent DMF.

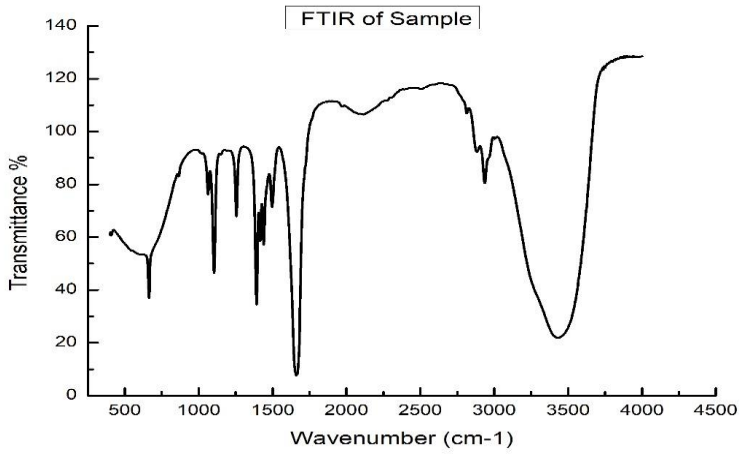

Fig.1 FTIR of as prepared sample

The FTIR response of the sample shows peaks corresponding at 667, 1112, 1258, 1393, 1450, 1509, 1673, 2941, 3445 cm^{-1}. On comparing with the reference response of bulk iron oxide, one peak just before 1000 cm^{-1} almost corresponds with that found in the sample's response. From the wavenumber range of 1000 to 3000 cm^{-1}, the curve matches almost exactly to that of the solvent DMF. The solvent poses to be larger in volume and the nanoparticles are suspended in the solvent can be taken to be almost negligible in comparison. In order to accurately verify the formation of 2D iron oxide we need to go for further analytical studies like Raman scattering, XRD, etc. To delve into the morphology of the nanoparticles formed, we went for its microstructural studies. Transmission Electron Microscopy (TEM) was done to obtain high resolution images and to see whether the desired planar structure was formed or not. From the TEM images below, we see that flat sheet like structures are formed. When compared to that of the reference HRTEM images of 2D iron oxide, the prepared sample shows clustered flakes

with a higher degree of opacity. This indicates that the thickness of the prepared sample is more than that what is desired for thin sheets of the reduced bulk iron oxide. The cluster of particles also indicates that the hydroxyl surface functional groups may still be present in the final product at large.

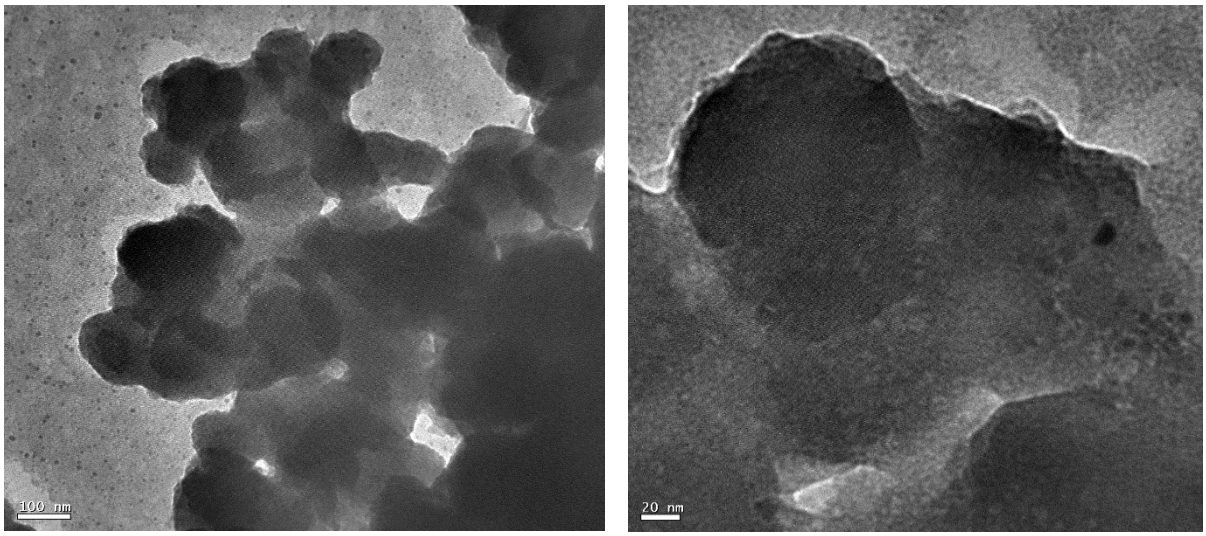

Fig. 2 TEM images of the sample at (a) 100 nm and (b) 20 nm magnification

The magnetic characterization needs to be carried out for the above system in order to determine the magnetic interaction present in the planar structure of iron oxide system. To get a comparative idea of the magnetic parameters, two systems of iron oxide of different morphology will be studied extensively. The above planar system will be compared with another prepared system of iron oxide. This latter system possesses a spherical structure and the final nanosytem has an assembly of such individual particles as shown below in Fig. 3. The difference in the arrangement of magnetic spins might give rise to interesting results which could be then correlated and supported with the SAXS and SANS data.

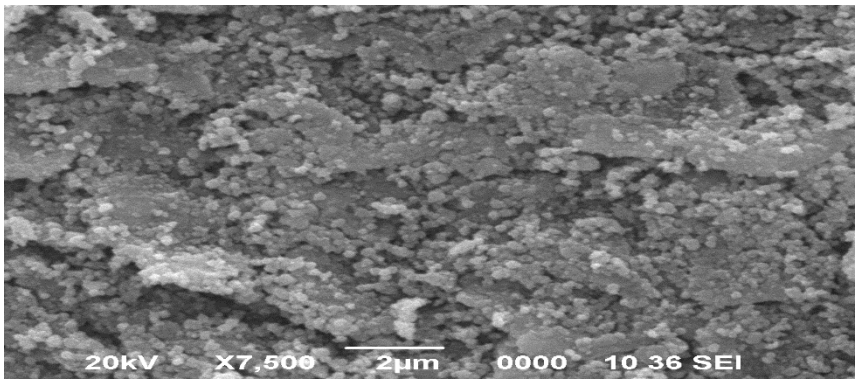

Fig. 3 SEM image of spherical iron oxide nanoparticles

(7) Date of 1st release of grant and total number of grants received so far:
31.03.2022, One grant received so far

(8) Name of the student (if funded) and her/his date of joining/leaving:
Sayoree Purakayastha, 25.08.2022

(9) Full details of publications arising out of the CRS project with title, name of all authors, and citations.
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