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"MICROWAVE ASSISTED ONE-POT SYNTHESIS OF Bis (5-BROMO-1H-INDOL-3YL)(4-ALKYLPHENYL) METHANE DERIVATIVES EMPLOYING $CuFe_2O_4$ NANOPARTICLE"

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KEYWORDS

$CuFe_2O_4$ nanoparticle, sol-gel method, Bis(indole) derivatives, EtOH solvent, ecofriendly synthesis etc.

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Abstract

In present work effective one pot and facile protocol was established for the series of Bis (5-bromo-1H-indol-3yl)(4-alkylphenyl) methane, Bromo indole and Substituted aromatic aldehydes, molecule reacted together in presence of synthesized $CuFe_2O_4$ nanoparticle to afford new derivatives of Bis(indole), the strategy works under mild circumstances and desired products obtained were pure enough without the need of chromatographic separation.

The Synthesized nanocatalyst analyzed by XRD, SEM & TEM which confirmed the successful formation of the $CuFe_2O_4$nanoparticle, this nanocatalysts exhibited easy preparation methods, environmentally friendly characteristics and excellent catalytic performance based on the obtained results, without the prepared $CuFe_2O_4$ catalyst the reaction route could not be completed, A number of transformations were performed with a catalytic loading in ethanol delivering the bis-indole derivative in good to yield, this obtained derivatives of Bis(indole) characterized by FT-IR, ^{1}HNMR, ^{13}CNMR and Mass spectroscopy.

This study demonstrated the novel synthesis of Bis(indole) derivatives using $CuFe_2O_4$ nanocatalyst and highlights the importance of nanocatalysts in environmentally sustainable organic synthesis.

INTRODUCTION:

Recently $CuFe_2O_4$ nanoparticles (NPs) have expanded attention due to their catalytic, magnetic, and electrical properties [1–5] , $CuFe_2O_4$ NPS have been extensively studied as effective catalysts in organic transformation, owing for it's redox qualities, thermal stability and magnetic separability, which allows easy recovery and reuse. Therefore, the present study highlights the preparation, applications and characterization of $CuFe_2O_4$ NPs in organic synthesis. $CuFe_2O_4$ NPs have emerged as a subject of extensive research interest largely due to their applications in catalysts, sensors, water treatment, photocatalysts, and air purification systems [6,7] $CuFe_2O_4$ MNPs confirmed excellent catalytic activities in several organic transformations, with the formation of propargylamine and triazoles [8], diarly ethers [9] bis(indolyl)methane [10] tetrazoles [11] dipyrimidines [12] and pyridines [13].

These studies showed that sol-gel and gel combustion technology looks as a capable choices, That's the reason so many researchers have recently adopted this method in their work, sol-gel preparation

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method is used for synthesis $CuFe_2O_4$ NPs in present study, We aimed to demonstrate the $CuFe_2O_4$ nanoparticles are low-cost catalyst, reusable, environmentally friendly, non-expensive and non-volatile[14] ,Bis(indole) derivatives are widely found in nature and the chemistry of Bis(indole) is one of the most interesting fields of research, Bis(indole) derivatives have been the topic of interest from synthetic and biological point of view.

The class of Bis(indole) compounds has received excellent attention in past few decade since they possess an excellent biological and pharmaceutical activities [15] , A series of recent studies refer to strategies for the synthesis of Bis(indole) derivatives with various medicinal properties such as antimicrobial, antiviral, anti-Alzheimer, etc.[16]These various biological pharmacological activities are b-glycosidase inhibitory [17], KAR-2 on store-operated calcium entry in human neutrophils [18] antiviral enantiomers [19] a cytotoxic [20] antiviral glycosidic [21] breast cancer cell proliferation via SIRT-p53 axis [22]anti-cancer chemo preventive agents [23] protein tyrosine phosphatase-1B inhibitory [24] anti-hyperlipidemic effects [25] etc.

There are nearly typical examples of catalysts used for the preparation of derivatives of Bis(indole) such as $Mg(ClO_4)_2$[26] MNPs@Cu nanocatalysts[27] Zirconium(**IV**) Chloride [28] and ZrOCl2.8H2O [29] ongoing research is directed towards the design of novel Bis(indole) frameworks, the development of efficient catalytic system, these methods often suffer from limitations including low product yields, reliance on toxic solvents, high operational costs, elevated reaction temperatures, and the need for specialized equipment, The application of $CuFe_2O_4$ nanoparticles in Bis(indole) synthesis provides various applications like short duration time, mild reaction conditions, improved yields and facial catalyst recovery using an external magnetic field.

Moreover, these nanoparticles promote green chemistry principles by reducing the need for hazardous reagents and minimizing waste generation. Hence the use of $CuFe_2O_4$ nanoparticles as a heterogeneous catalyst represents a promising and sustainable approach for the synthesis of biologically important indole derivatives aligning with the current demand for eco-friendly and efficient synthetic methodologies.

EXPERIMENTAL

MATERIALS AND METHODS:

All the chemicals Copper nitrate, Ferric nitrate, Citric acid, ammonium solution, Aromatic aldehyde, sub. indole, ethanol etc. required reagents and chemicals were acquired from Sigma Aldrich and Merck private limited, X-ray diffraction (XRD) was recorded in Bruker Kappa Apex., Transmission Electron Microscope (TEM) images of nanoparticles were obtained using FEI Tecnai T20 S-Twin instrument, Scanning electron microscope (SEM) images of the nanoparticles were obtained using FEI-APREO CHEMISEM Instrument, FT-IR Spectra were recorded in Shimadzu FT-IR 8400 Spectrometer. ^{1}H NMR and ^{13}C NMR spectra were recorded in Bruker Avance Neo 500 (DRX)MHz, NMR spectrometer, Mass measurements were performed on Bruker impact HD Q-TOF analyzer in the ESI mode. After completing the reaction Melting points determination in Buchi melting point apparatus B-540 B.V.CHI Monitoring of reactions were performed using TLC (Merk kieselgel 60 0.20 mm layer UV254).

GENERAL PROCEDURES:

1) Synthesis of $CuFe_2O_4$ Nanoparticles

$CuFe_2O_4$ nanoparticles were synthesized by the sol-gel method, stoichiometric amount of nitrate salts that are Iron nitrate Fe $(NO_3)_3.9H_2O$(0.02ml), Copper nitrate $(NO_3)_2.3H_2O$(0.01ml), and citric acid (0.03ml) were dissolved in (50ml) distilled water. Then, alkaline pH was adjusted by ammonia solution. This solution heated at 80°C with constant stirring until viscous gel formed, gel was heated in an oven at 120°C, then dried product formed which was ground into a fine powder using mortar and pestle then calcined in muffle furnace at 850°C for 4 h after cooling at RT, a black crystalline $CuFe_2O_4$ nanopowder obtained then further characterization XRD, SEM & TEM done.

2) Synthesis of Bis (5-bromo-1H-indol-3yl)(4-alkylphenyl)methane

A mixture of 5-bromo-1H-indole (2mmol), Sub. Aldehyde (1mmol) and $CuFe_2O_4$ nanoparticle (20 mg) were added followed by 5 ml of Ethanol as the reaction medium, the reaction mixture was irradiated in a microwave reactor at 80^0 c for 20-25 min, the progress of reaction was monitored by thin layer chromatography (TLC) using hexane: ethyl acetate (7:3) as mobile phase. after completion the reaction mixture was allowed to cool at RT. The $CuFe_2O_4$ nanocatalyst was separated using an external magnet, washed with ethanol, dried and reused in next reaction cycles. After separation the crude product was washed with cold water and recrystallized from ethanol to get product, after that further analysis such as

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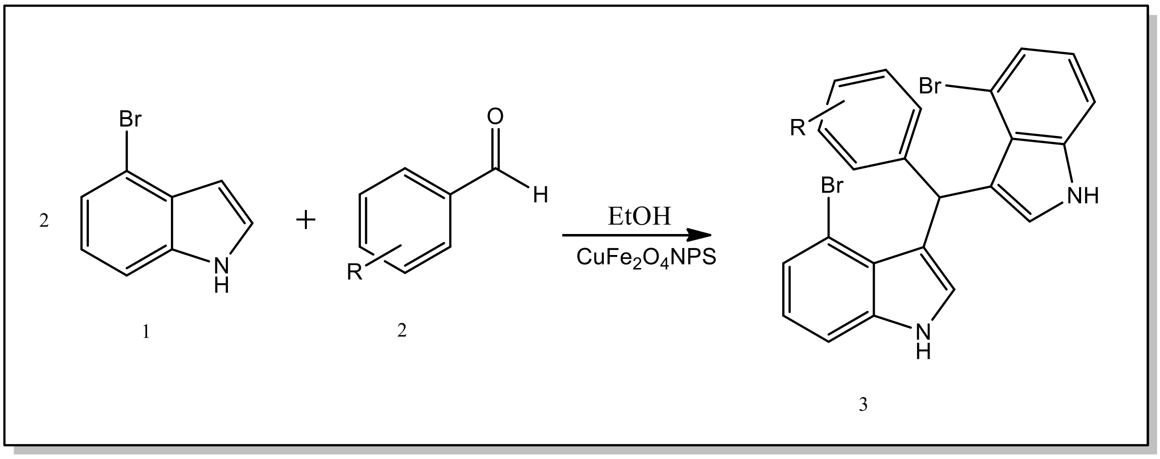
IR,[1]HNMR,[13]CMR & Mass spectroscopy done.

RESULTS AND DISCUSSION:

In search of the best experimental reaction conditions, we used $CuFe_2O_4$ as a catalyst represent an efficient, economical and environmentally friendly magnetic nanocatalyst for the synthesis of Bis (5-bromo-1H-indol-3yl)(4-alkylphenyl)methane and it's derivatives using microwave irradiation, in this process the use of hazardous organic solvents is minimized and helps in environment protection from pollutions. Whereas, most of the procedures developed for this reaction were reported by using hazardous organic solvents. Thus, that the process developed is greener as compare to the reported ones.



Scheme 1; Synthesis of Bis (5-bromo-1H-indol-3yl)(4-alkylphenyl)methane using $CuFe_2O_4$ nanocatalyst.

To accomplish the same transformation the reaction was subsequently performed under microwave irradiation while maintaining the identical reaction conditions used in the conventional procedure. The performance of the microwave assisted protocol was then evaluated against the conventional method. Although both methods afforded the desired products, the microwave assisted approach demonstrated superior efficiency by providing higher product yields (80-92%) and considerably reducing the reaction time to 20-30 min. the comparison between the conventional and microwave methods is summarized in following **Table no.1.**

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Table: 1-Synthesis of Bis (5-bromo -1H-indol-3yl)(4-alkylphenyl) methane using $CuFe_2O_4$ nanocatalyst.

Sr.no.	Entry	R	Conventional mode, Time(h)	Yield%	Microwave mode, Time(min)	Yield%
1	AK1	4-Cl	5	79	25	92
2	AK2	3-NO_2	5	82	25	90
3	AK3	4-NO_2	4	75	20	80
4	AK4	4-Ome	5	69	30	85
5	AK5	4-OH	4	70	35	89

Synthesis of Bis (5-bromo-1H-indol-3yl)(4-alkylphenyl)methane derivatives was investigated using $CuFe_2O_4$ nanoparticle under microwave irradiation. various reaction parameters including catalyst loading, reaction time were optimized amount of $CuFe_2O_4$ nanoparticles afforded the desired products in excellent yields within a short reaction time, operational simplicity and straightforward catalyst recovery compared with this previously reported catalyst, $CuFe_2O_4$ nanocatalyst gaves good results shown in

Table no. 2.

Table no. 2 : Synthesis of Bis (5-bromo -1H-indol-3yl)(4-alkylphenyl)methane using $CuFe_2O_4$ nanocatalyst compared with other catalyst.

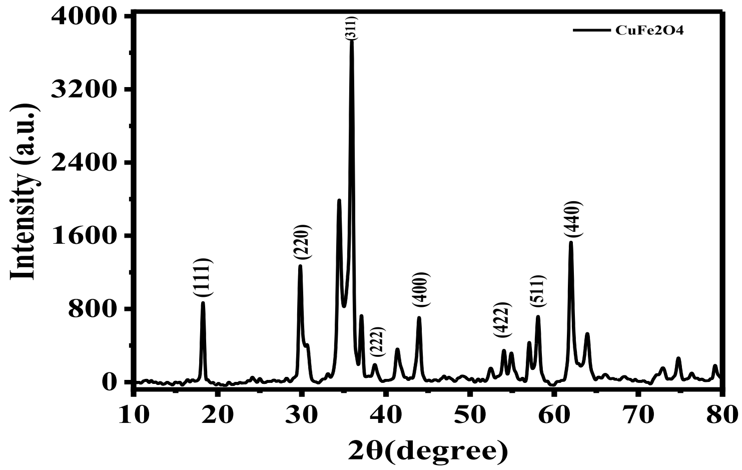
Sr.no.	Name of catalyst	Time (min.)	Yield %	Reference
1	$Mg(ClO_4)_2$	45	88	[26]
2	MNPs@Cu	40	80	[27]
3	Zirconium (IV) Chloride	35	87	[28]
4	$ZrOCl_2.8H_2O$	180	85	[29]
5	**$CuFe_2O_4$**	**25**	**92**	**Present work**

Characterization of synthesized $CuFe_2O_4$ nanoparticle.

1] **XRD (X- ray diffraction)**

The XRD pattern exhibits characteristic diffraction peaks at 2θ degree 18.5,30.2,35.4,43.2,57.0 and 62.5 which are indexed to the (111), (220), (311), (400), (511) and (440) planes of a cubic spinel structure. The (311) reflection observed at 35.4 intense peak indicating that the synthesized $CuFe_2O_4$ nanoparticles possess high crystallinity. The diffraction peaks closely match the standard JCPDS Card for $CuFe_2O_4$ confirms the successful formation of crystalline Nanoparticle.

Fig 1; XRD of synthesized $CuFe_2O_4$ nanoparticle



2] **SEM (Scanning Electron Microscope)**

SEM micrograph exhibits that the synthesized $CuFe_2O_4$ nanoparticles possess a quasi-spherical morphology with noticeable agglomeration. The particle size ranges from approximately 60 to 100 nm with an average size around 80 nm. The observed aggregation is attributed to magnetic interactions and high surface energy of the nanoparticles. The porous and rough surface area which is advantageous for catalytic and biological applications.

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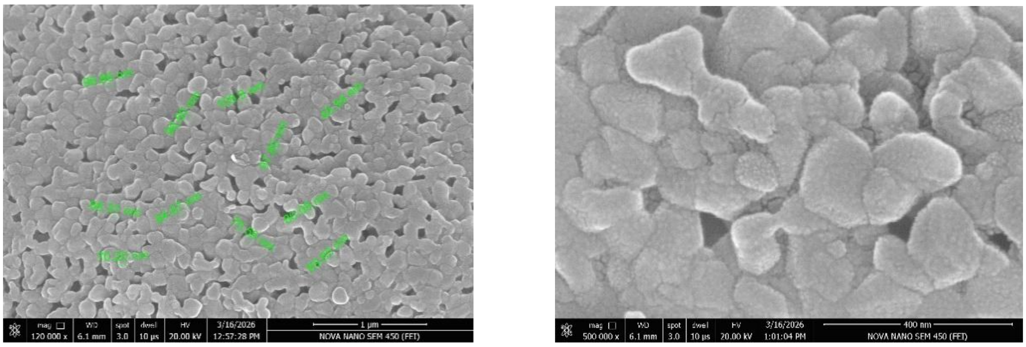

Fig 2: SEM images of synthesized $CuFe_2O_4$ nanoparticl

3) **TEM (Transmission Electron Microscope):**

The TEM image reveals agglomerated $CuFe_2O_4$ nanoparticle with irregular to quasi-spherical morphology and particle sizes range of 20-60 nm. The SAED pattern exhibits a combination of discrete diffraction spots and concentric rings, indicating a polycrystalline nature with partially oriented crystallites. The diffraction rings are indexed to the (220), (311), (400), (422), (511) and (440) planes of the cubic spinel structure, confirming the formation of phase pure $CuFe_2O_4$ nanoparticle with good crystallinity.

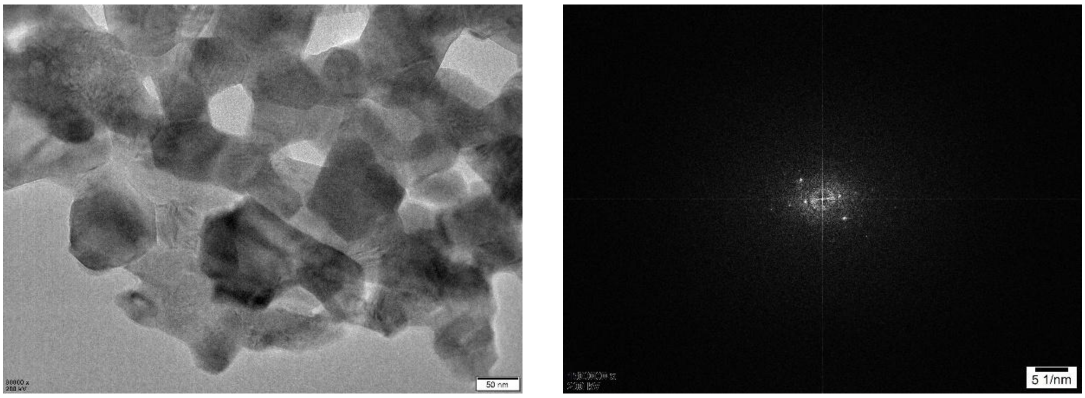

Fig 3; TEM images of synthesized $CuFe_2O_4$ nanoparticle

SPECTRAL DATA OF COMPOUNDS (AK1, AK2, AK3, AK4,AK5)

1] (Ak1) Bis (5-bromo-1H-indol-3yl)(4-chlorophenyl)methane

FT-IRSpectrum(vmax,cm^{-1});3419(NH,indole),3108(ArH),2916,2848 (CH),1607-1517(Ar-C=C), 1380-1250(C-N),740-690(C-Br),690-620(C-Cl).

^{1}HNMR(DMSO-d_6,δppm);11.10(br s,2H,NH),7.44(d,2H),7.36-7.29(m,4H),7.24(d,2H),717-7.09(m,4H,Ar-H),6.99(d,2H,Ar-H),5.99(s,1H,CH)

^{13}CNMR Spectrum (DMSO,$d_6$ppm; 145-137(Ar-C-N,C-Br)),132-126(Ar-C,indole),125-119(Ar-C),118-111(indole Ar-C),110-104(C-Br),40(-CH)

Mass spectrum: ***m/z*** = 515.56 g/mol.

2] AK2; Bis (5-bromo-1H-indol-3yl)(3-Nitrophenyl)methane

FT-IR Spectrum:3418(N-H),3060(C-H),2925(C=C),1604,1525,1458(Ar C-C),1348,1265(C-N),1092(C-N),825(ArC-H),620(C-Br).

^{1}HNMR:11.16(s,2H,NH),8.10(d,1H,Ar-H),8.02(dd,1H,Ar-H),7.85(d,1H,Ar-H),7.72(t,1H,Ar-H),7.59-6.95(m,indoleAr-H),6.0(s,1H,CH).

^{13}CNMR:147.6,139.8,134.5,132.0,130,127.4,125.9,124.7,123.6,122,121,120.5,118.8,115.8,112,110.5,39.6.

Mass Spectrum: 529 g/mol

3]AK3; Bis (5-bromo-1H-indol-3yl)(4-Nitrophenyl)methane

FT-IR Spectrum; 3416(N-H),3040(Ar-C-H),1620(C=C),1518,1340(NO_2),1245(C-N),620 (C-Br)

^{1}HNMR(DMSO-d_6,δppm):11.10(s,2H,NH),8.18(d,2H,Ar-H),7.36(2H,Ar-H),6.88-7.79(m,10H,Ar-H),5.88(s,1H,CH).

^{13}CNMR(DMSO,$d_6$ppm):145,134.6,131, 126.8,125.4,122.6,117.115.4,112,108,42.4.

Mass Spectrum: ***m/z*** = 524.60 g/mol.

4] AK4 Bis (5-bromo-1H-indol-3yl)(4-methoxyphenyl)methane

FT-IRSpectrum:3288(NH),3145(Ar-C-H),2840(OCH3),1600(C=C),1190(Ar-O-CH3),1040(C-O-C),630(C-Br).

^{1}HNMR(DMSO-d6, δ ppm):11.05(br s,2H,NH),7.30(d,2H,Ar-H),6.80-7.58(m,10H,Ar-H),6.70(d,2H,Ar-H)5.85(s,1H,CH),3.80(s,3H,OCH_3).

^{13}CNMR(DMSO-d6,δppm;155,132.4,128.6,125,123,118.5,113.2,109.4,50.5,40.6.

Mass Spectrum *m/z;* 512 g/mol

5] AK5 Bis (5-bromo-1H-indol-3yl)(4-Hydroxyphenyl)methane

FT-IR Spectrum;3407(O-H),3328(N-H),3056(Ar C-H),2920(C-H),1620(Ar C-H),1450(Ar C=C),1262(Ph C-O),1160(C-N),812(C-Br),740(C-H).

^{1}HNMR(DMSO-d6,δppm); 11.0(br,s,2H,2NH),9.25(s,1H),7.60(s,2H),7.46(d,2H), 7.10(d,2H),7.04(d,2H),6.66(d,2H),6.05(s,1H).

^{13}CNMR(DMSO-d6, δppm;154.05,134.4,132,130.8,128,126.8,124.6,122,120,118.2,115, 113.8,111.4,40.08

Mass Spectrum *m/z;* 497g/mol.

RECYCLABILITY OF $CuFe_2O_4$ NPs :

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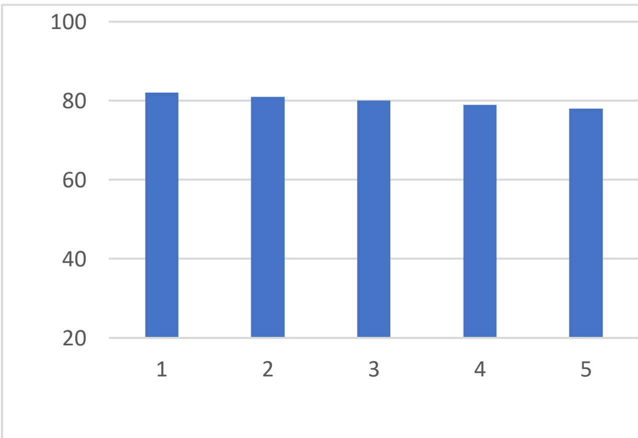
In order to explore the recyclability of the catalyst, the $CuFe_2O_4$ nanoparticles were used as catalyst for the same reaction repeatedly and the change in their catalytic activity was studied. The relation between the number of cycles of the reaction and the catalytic activity in terms of yield of the products is presented, It was found that copper nanoparticles could be reused for five cycles with negligible loss of their activity shown in (Fig.4), The possibility of recovery and reuse of several studies have demonstrated that $CuFe_2O_4$ NPs retain high catalytic activity, the minor loss in catalytic performance is generally attributed to small catalyst losses during washing, the recyclability of $CuFe_2O_4$ nanoparticles contributes significantly to the principles of green chemistry by reducing catalyst consumption, lowering operational costs, minimizing waste generation and enabling catalytic processes. These characteristics makes $CuFe_2O_4$ an attractive catalyst for an applications in heterocyclic compound synthesis.

Fig 4; Recyclability of $CuFe_2O_4$



CONCLUSION:

In summary, we have developed efficient, rapid and environmentally friendly Microwave assisted protocol was successfully developed for the synthesis of Bis (5-bromo-1H-indol-3yl)(4-alkylphenyl)methane derivatives using $CuFe_2O_4$ nanocatalyst, the microwave assisted approach significantly reduced the reaction time while providing excellent yields under mild reaction conditions. The catalytic activity of $CuFe_2O_4$ nanoparticles can be attributed to their high surface area, abundant active sites and easy magnetic separation making the process simple, economical and sustainable. The synthesized $CuFe_2O_4$ nanoparticles were successfully characterized by XRD, SEM & TEM Confirming their crystalline spinel structure, morphology and nanoscale particle size. The structure of the prepared Bis (5-bromo-1H-indol-3yl) (4-alkylphenyl) methane derivatives were confirmed by FT-IR,^{1}HNMR ^{13}CNMR and Mass spectroscopy which gives good results. The developed synthesis provides promising approach for the synthesis of biologically important Bis(indole) compounds and a valuable contribution to green organic synthesis.

DISCLOSURE STATEMENT:

The authors declares that there is no conflict of interest.

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