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ALOE VERA EXTRACT STABILISED NANO SILVER- REDUCED GRAPHENE OXIDE NANO COMPOSITES AS VISIBLE LIGHT PHOTOCATALYST FOR OXIDATIVE DEGRADATIONS OF RANITIDINE HYDROCHLORIDE AND SODIUM DICLOFENAC IN AQUEOUS MEDIUM: A KINETIC STUDY

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Keywords:

Aloe vera stabilized silver nano particles, Reduced graphene oxide nanocomposites, Visible light drug degradations, Oxidative mineralization, Ranitidine hydrochloride, Sodium diclofenac

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ABSTRACT: Nano composites of reduced graphene oxide (rGO) and silver nanoparticles (AgNps) were prepared in this work, adopting green reaction conditions. Aloe vera plant extract was used as the stabilizing agent in the AgNps system and graphite precursor has been chosen for rGO preparation adopting mild hydrothermal and sonication methods. AgNps and AgNp–rGO Nano composites are characterized using UV-VIS SPR; powder XRD, HR-TEM and FE-SEM measurements. The catalytic ability of AgNp–rGO nano composites in the as-synthesized form has been confirmed by following the oxidative degradation of the two therapeutically potential drugs such as sodium diclofenac (NaDF) and ranitidine hydrochloride (RHCl) under visible light irradiation in aqueous medium at 25°C. Reaction kinetic parameters and the oxidant peroxo monosulfate ($Na_2S_2O_5$) effect in presence of AgNp–rGO nano-composites as the catalyst under visible light irradiation exhibited remarkable catalytic behavior in presence of visible light for the degradation of the two drug both in presence and absence of oxidant. However presence of oxidant enhanced the speed of drug degradations with AgNp–rGO nano composites and visible light. Among the two studied drugs, RHCl degraded quickly and also, efficiently mineralized compared to NaDF. Degradation or carcinogen NDMA was not observed and the two drugs are mineralized to near completion. The results are favourable towards the use of AgNp–rGO nano composite as a potential catalyst in AgNPs employed in the treatment of drug polluted waste waters.

INTRODUCTION: Immobilisation of transition metal nanoparticles into synergetic and extensively conjugated carboneous supports like graphene are exploited widely in reaction catalysis, photoactivity and electronic sensing cum conduction applications in current science [1-6].

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Recently research reports exists on graphene and its functionalized forms like graphene oxide (GO) and reduced graphene oxide(rGO) as suitable and strong supports for deposition of metal nano particles, resulting in multi-functional nano composites [7-10].

Among many metal nano particles, sliver nano particles (AgNps) exhibit potential photo catalytic activity with photo sensing applications in and UV-visible radiations ranges [11-15]. In this work, AgNps are prepared in a green pathway [16-17] using Aloe vera plant as the stabilising agent instead of

synthetic polymeric substrates. Numerous biomolecules present in plant extract function as both reducing and stabilising agent during the synthesis of AgNps [18-21]. The as synthesised AgNps are characterized by UV-VIS SPR, and powder XRD measurement, using graphite powder, GO was synthesized adopting mild, and eco-friendlier hydro thermal process conditions followed by step wise GO formations and AgNps deposition. By doing so, literature reported procedures with minor modifications are adopted [22, 23]. The designed AgNps-rGO nano composites are subjected to HR-TEM and FE-SEM analysis. The applicability of AgNps-rGO nano composites as efficient photo sensing and photo catalytic agent in industrial waste waters treatment has been attempted on medicinal drug polluted waters. Environmental aquatic sources suffer severe damages such as reduced light penetration, lesser solubility of oxygen and other gases, and threatening of life systems etc, mainly due to pharmaceutical waste water effluents [24-26]. Pharmaceutical drugs possess synthetic complex organic structures that are difficult to self-degrade. In this work two potential therapeutically used drugs such as Ranitidine hydrochloride (RHCl) and Sodiumdiclofenac (NaDF) are chosen among the many existing drugs that cause severe water pollutions. RHCl is a histamine H_2- antagonist widely applied in the treatment of peptic ulcers and gastro oesophageal reflux diseases [27, 28]. RHCl prolonged leakage into environmental water bodies cause eco – toxicity to aqua life systems and another dangerous risk of RHCl polluted waters, may be the formation of N-nitrosodimethylamine (NDMA)which is a potential carcinogen and its removal from waste waters is mostly weak and poor [29, 30]. NaDF, belongs to a non-steroidal anti-inflammatory drug (NSAID) which is widely used as analgesic, antiarthritic and antirhenmatic [31, 32]. NaDF polluted water treatment needs efficient AOPs, due to its stability and slow removal of organic matter.

In **Fig. 1** molecular structures of RHCl and NaDF are given. Photo catalytic oxidative degradation processes under UV or visible light irradiations involving designed nano composites materials may be used in the treatment of drugs polluted waters.

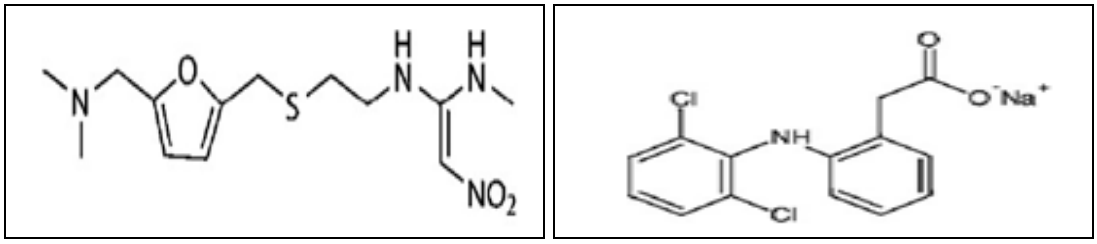


FIG. 1: MOLECULAR STRUCTURES OF RHCL AND NADF

In this work, AgNps-rGO nano composites are used as the photo catalyst to degrade the two chosen drugs RHCl and NaDF in presence of an oxidant sodium peroxo mono sulphate ($Na_2S_2O_5$) under visible light irradiation at 25°C. AgNps, due to the narrow band gap, in the presence of visible light and aerobic conditions, produce reactive oxygen species (ROS) such as OH and O_2 which are released in aqueous medium. These ROS, tend to react non selectively with drug molecules and mineralize completely in rapid pathways. Presence of rGO with AgNps, suppresses the photo electron and whole recombination and deactivation of OH radicals via forming H_2O_2 steps. Henceforth, adopting pseudo first order reaction conditions, the photo oxidative degradations are followed by recording the UV-spectra absorbance decrease with the time of progress of the reactions. The wave length maxima for RHCl is 313 nm and for NaDF is 280 nm respectively, are fixed for absorbance-time data of small aliquots drawn out at intervals of time. Also, the overall pseudo first order rate co-efficient (k) values and % mineralisation of the two drugs are found out. The results indicate that AgNps-rGO nano composite serves as an efficient photo catalyst in AOP for facile and complete mineralization of RHCl and NaDF in visible light.

EXPERIMENTAL: Sodium hydroxide pellets, silver nitrate ($AgNO_3$), sodium peroxo-monosuphate, graphite powder with particle size < 50 µm, 98% sulphuric acid, potassium permanganate and potassium nitrate were procured from Merck India. Ranitidine hydrochloride and Sodium diclofenac with 99% purity were purchased

from Sigma-Aldrich. Fresh triply distilled water was used in all solution preparations.

Synthesis of Ag Nano particles (AgNps): Fresh and sliced Aloe Vera pieces (40gms) were ground to a fine paste adding 50 ml of NaOH solution at pH = 6.0. The slimy solution was filtered and the clear filtrate was used 20 ml of 1 mM Ag NO_3 solution prepared in 0.1M KNO_3 solution was taken and 50 ml of Aloe vera extract solution was added drop wise maintaining 40°C as the solution temperature with constant stirring for 90 minutes period of time. Formation of a lemon yellow coloured solution indicated the formation of AgNp.

Repeated ultra-centrifugations and washings of residue resulted in AgNp in wet condition. This procedure was repeated several times and the AgNp suspension in dilute (0.01M) KNO_3 solution was stored for size characterizations as reported earlier [33, 34, 35]. UV-Visible, SPR recorded showed a prominent peak at 405 nm confirming the presence of AgNp. Solid AgNp were collected after vacuum drying and stored in N_2 purged dark container.

Graphene oxide was synthesized from Graphite powder through improved Hummer's method. The procedure followed being 30 ml of 9:1 (V/V) mixture of concentrated H_2SO_4 and H_3PO_4 was added to 1.5g of graphite powder and 9.0g of $KMnO_4$ with constant stirring for 12 hrs at 45°C. The reaction mixture was cooled to 25°C and added 5ml of 30% H_2O_2 to convert MnO_2 residue to soluble sulphate form. Excess (250ml) triple distilled water was added and stirred for1hr at 70°C. After cooling, the mixture was ultra-centrifuged several times with repeated washings. The final solid was coagulated with ether solvent and dark brown crystalline GO was vacuum dried and stored in dark and N_2 purged container.

AgNp-rGO nano composites are prepared by carrying out a procedure with minor modification to the reported one 33. 50 mg powder of GO was dispersed into 100 ml of triple distilled water and ultrasonicated for 1 hr to form yellowish brown exfoliated GO sheets. To this slurry, 30 mg of AgNps mixed in 50ml of 0.01 M KNO_3 was added slowly under constant stirring at 60°C as the medium temperature of 1hr time period. The black precipitate (AgNp-rGO) obtained was washed several times to remove any unbound AgNps and other impurities. Vacuum dried AgNp-rGO nano composites are stored for further use.

Spectra Measurements and Size Characterization: All UV-visible spectra were recorded on a Schimadzu (UV-1650 Pc) spectrometer fitted with thermostat for temperature control. Powder XRD patterns were obtained using Bruker diffractometer D8 Advance with CuKα radiation. HR-TEM measurements were carried out using TEM Hitachi Technai G20 and for FE-SEM, Hitachi FESEM S4800 microscopes respectively.

Reaction Photo Catalysis: 50 ml of 1 mm drug solution was left in dark for 3hrs to reach equilibrium and loaded into glass Pyrex cylindrical photo reactor equipped with continuous N2 or air purging, magnetic stirrer and a temperature controller. The photo reactor was irradiated by 20 series white light LEDs (nominal power 6W) with wave length emission in the visible range 400-800 mm. 50ml of 0.1M $Na_2S_2O_5$ cool solution was added and N2 purged after each addition of any component into the reaction vessel.

After the addition of AgNp-rGO nano composite catalyst, small aliquots of the drug solution was drawn out at regular intervals of time and subjected to UV spectra measurements. The completion of the reaction was known from the decrease in the absorbance of peak maximum to zero value. Pseudo first order conditions on the compositions of the drug, catalyst and oxidant are maintained, and the pseudo first order rate coefficient values are determined from the absorbance time variance data.

RESULTS AND DISCUSSION: The UV visible spectrum and Powder XRD of Aloe Vera stabilized AgNp and AgNp-rGO is given **Fig. 2** and **Fig. 3** respectively.

The powder PXRD patterns of AgNp confirm with the JCPDS file no 04-0783. Using Scherrer equation, the size range of the AgNp has been found within 12$\pm$ 2.0 nm. FE – SEM and HR – TEM micro images of AgNp– rGO nano composites are shown in **Fig. 4** and **Fig. 5** respectively. In the SEM nano image, layers of rGO and AgNp depositions on the surface are observed.

Similar AgNps decoration on rGO surface are detected in HR – TEM images and the average size value of AgNps are found to be 16±1.0 nm indicating a marginal increase in the average size of AgNp due to surface interactions with rGO nano composites [33].

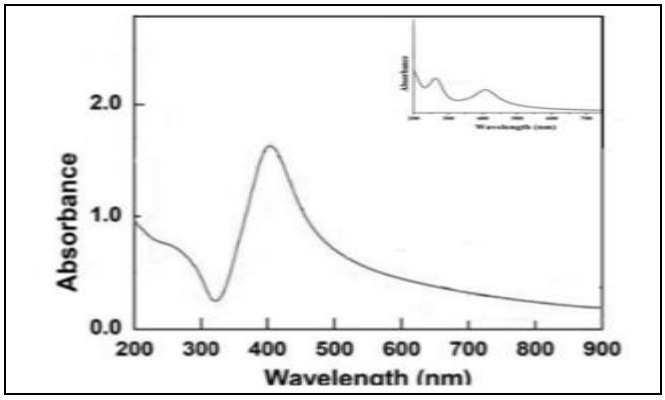


FIG. 2: UV-VISIBLE SPR SPECTRA OF AgNp WITH INSET FIGURE OF AgNp-rGO

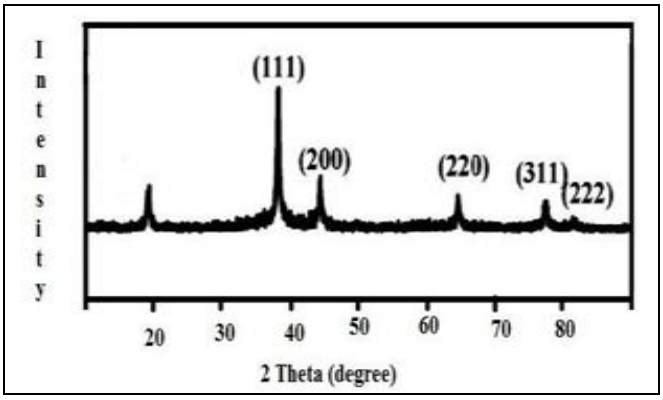


FIG. 3: POWDER XRD OF ALOE VERA AgNp-rGO

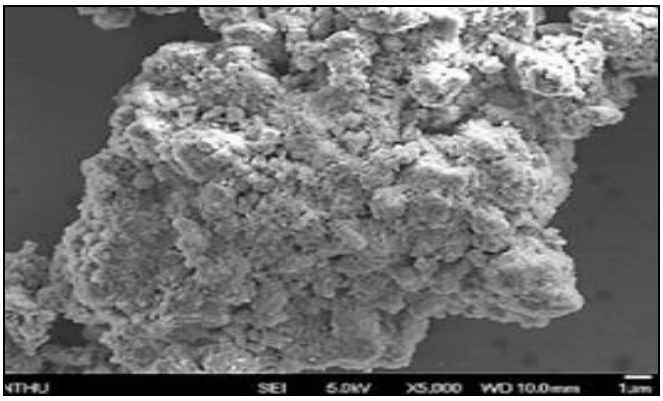


FIG. 4: FE-SEM IMAGES OF AgNp-Rgo NANO COMPOSITES

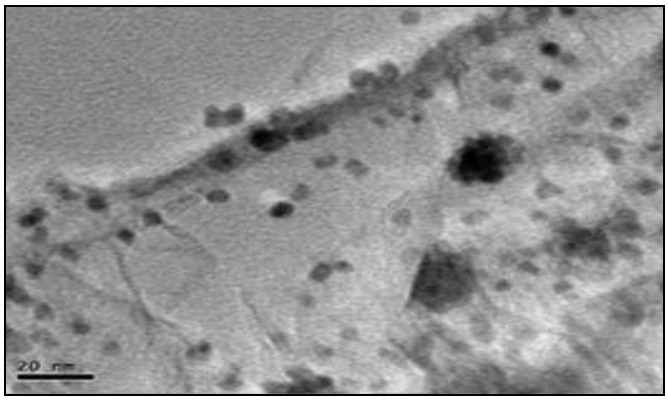


FIG. 5: HR-TEM MICRO PHOTO OF AgNp-rGO NANO COMPOSITES

In order to ascertain the photo catalytic behaviour of the fabricated AgNp–rGO nano composites prepared in green pathways, degradations of RHCl and NaDF dyes are studied separately and the followings alient observations are found.

The degradation of the drug started up only upon the start of visible light irradiation in the presence of nano composites and drug degradation oxidant. Also, the drug degradation was extremely slow in the absence of light but even in the presence of Ag NPs – rGO nano composite and the oxidant. In the absence of the oxidant but in the presence of nano composites and visible light, the dye decolouration was again found slow and took more than 8 hrs for complete degradation. Thus to sum up the degradation was faster only in the presence of light, oxidant and catalyst. The absorbance of the wave length maximum peak in the UV spectra for each of the drug was found to decrease with increase in the time of progress of the oxidation. In **Fig. 6** and **Fig. 7**, such time variances in the spectra are shown for RHCl and NaDF drugs respectively. The corresponding absorbance versus time plots for the two drugs are shown in **Fig. 8(A)** and **8(B).** The extent of absorbance decrease with time has been found to be more for RHCl than NaDF dye degradations.

These data are used for the kinetic plots from which the overall pseudo first order rate coefficient values are determined. These are shown in **Fig. 8(A)** and **8(B), Fig 9(A)** and **9(B)** respectively. The completion of the reaction was indicated from the absorbance values reaching zero value. The nanocomposite catalysts were recovered by filtration and washed repeatedly followed by ultra-centrifugation. The catalyst residue is vacuum dried and as well as stored in dark for 24 hrs to attain equilibrium. The catalyst activity of AgNp-rGO nano composite was tested by repeating, the dye degradation procedures.

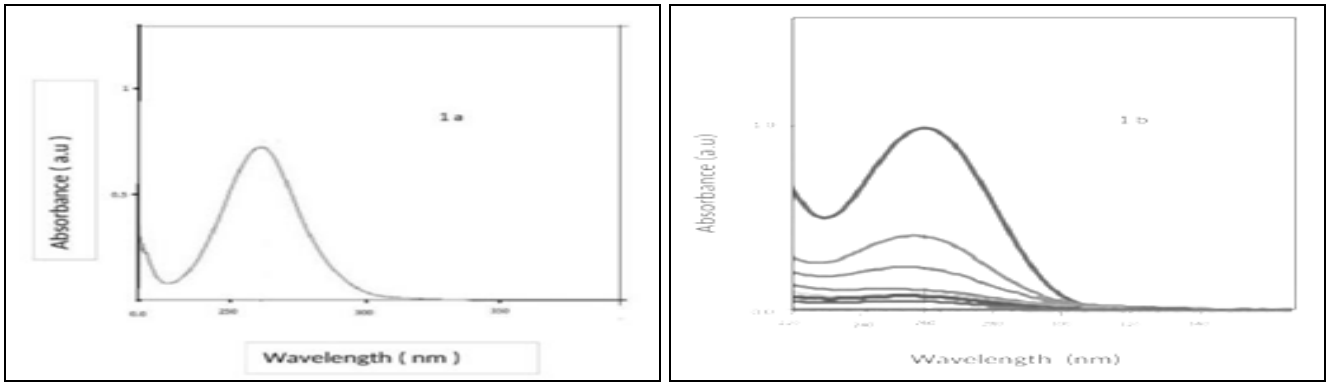


FIG. 6: VISIBLE SPECTRA WITH TIME VARIANCE OF NADF IN AQUEOUS MEDIUM IN HE PRESENCE OF AGNP-RGO NANO COMPOSITES AND PEROXO MONOSULFATE UNDER VISIBLE LIGHT IRRADIATION AT 25°C

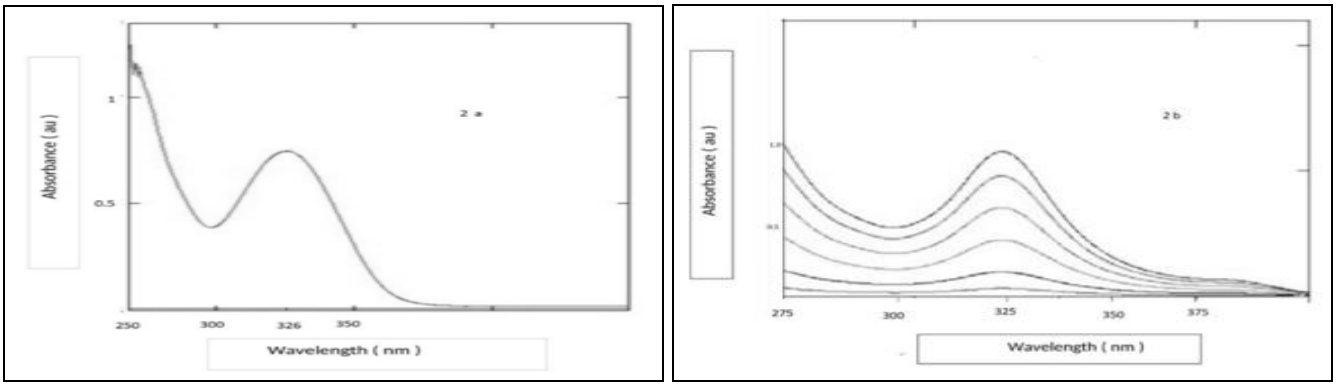


FIG. 7: VISIBLE SPECTRA WITH THE TIME VARIANCE OF RHCL IN AQUEOUS MEDIUM IN PRESENCE OF AGNP-RGO NANO COMPOSITES AND S_2O_5 UNDER VISIBLE LIGHT IRRADIATION AT 25°C

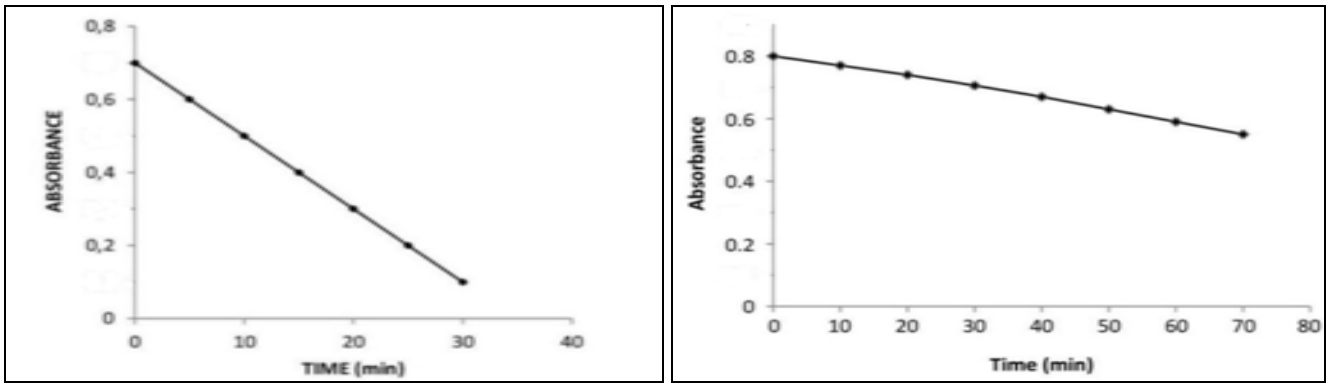


FIG. 8: ABSORBANCE VARIATION WITH TIME PLOTS FOR (A) RHCL AND (B) NADF PHOTO DEGRADATION UNDER VISIBLE LIGHT IRRADIATIONS AND AGNP-RGO NANOCOMPOSITE CATALYS WITH $S_2O_5^{2-}$ OXIDANT AT 25°C

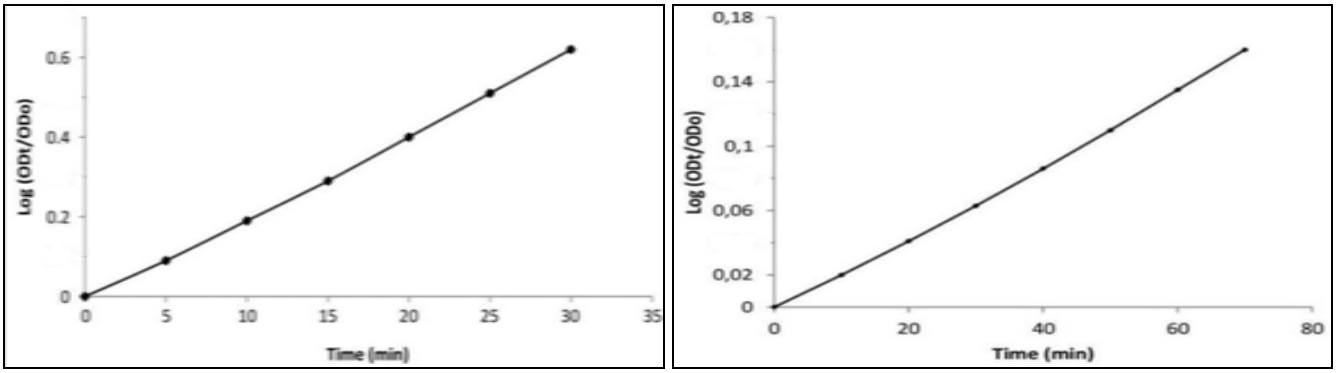
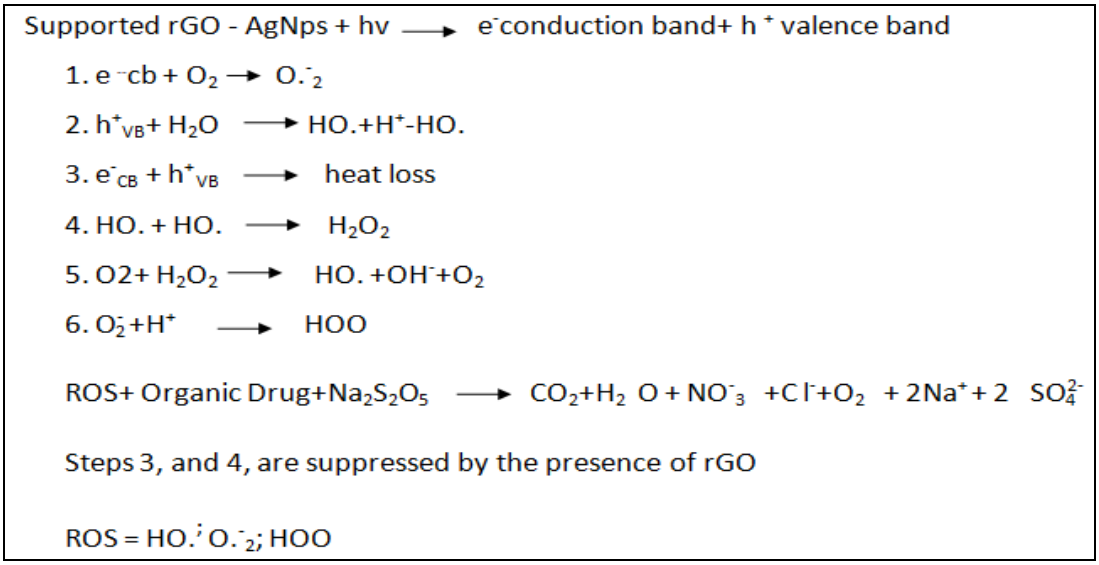


FIG. 9: PSEUDO FIRST ORDER KINETIC PLOTS FOR RATE COEFFICIENT DETERMINATION OF (A) RHCL AND (B) NADF PHOTO DEGRADATIONS UNDER VISIBLE LIGHT AND AGNP-RGO NANO COMPOSITE CATALYST AND $S_2O_5^{2-}$ OXIDANT AT 25°C

However, the drug degradation took hours, indicating that the recycled nano composites have lost the catalytic activity significantly. This may be attributed to the tarnishing of Ag metal surfaces by air oxygen. The reaction filtrate was analysed for the drug mineralization. In **Table 1** the kinetic parameters including the overall rate co efficient values for the pseudo first order reaction conditions and % mineralization of the drugs are put forth. The data indicate that RHCl degraded faster than NaDF in the visible irradiation and AgNp–rGO nano composite surface catalyses RHCl molecules more rapidly than NaDF molecules in the presence of $S_2O^{2-}_5$ oxidant. The plausible photo oxidation pathways of the two drugs are given in **Fig. 10**. Presence of Ag nanoparticles on the rGO matrix enhances the absorption of visible light, due to the strong SPR and facilitates the electron transfers as well as the H atoms transfers from the drug and the oxidant molecules. The rGO being a good electron acceptor and excellent charge transporter with excess π electrons facilitates the anchoring of the drug molecules suitable for e^- and H transfers. This contributes to the rate determining step .In consequence, cascadic mineralization of the organic drug molecules with CO_2 evolution wherever occured was detected. Also, no trace of NDMA was detected in RHCl degradations. These steps are essential in the water treatment by AOP during drug leakages into aquatic sources.

TABLE 1: THE OVERALL PSEUDO FIRST ORDER RATE COEFFICIENT (KX10^{-2} SEC^{-1}) VALUES FOR THE OXIDATION REACTIONS OF RHCL AND NADF USING PEROXOMONOSULFATE AND AGNP-RGO AS PHOTO CATALYST AT 25°C

Substrate	k	$t_{½}$ (min)	% mineralisation
RHCl	5.85	0.18	98.0%
NaDF	0.416	2.30	96%



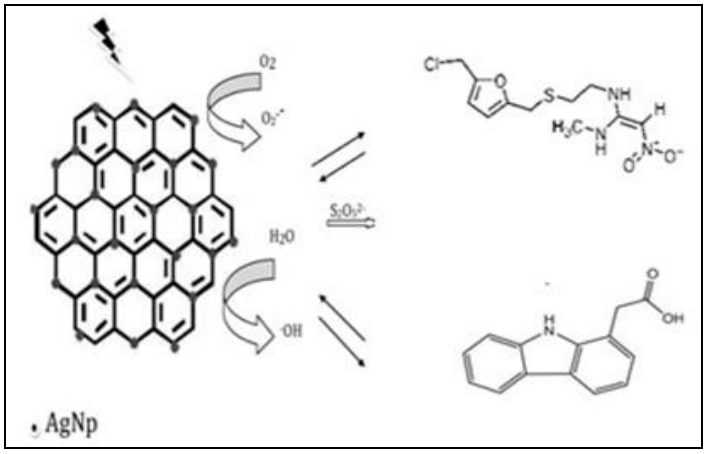


FIG. 10: PLAUSIBLE PHOTO MECHANISM OF OXIDATION OF DRUGS WITH S2O5^{2-} OXIDANT UNDER VISIBLE LIGHT IRRADIATION

CONCLUSION: AgNps synthesized by a green pathway using Aloe vera plant extract resulted in stable nano particles which are conveniently deposited on to rGO surface resulting in AgNps-rGO nano composites. The fabricated nano composite material is used as the photo catalyst for the oxidative degradation of the two popular drugs RHCl and NaDF under visible light in the presence of peroxomonosulfate oxidant. Remarkable drug degradations took place and RHCl degraded more rapidly than NaDF along with near complete drug mineralisations. Also, no trace of NDMA was found in RHCl degradation products. These results indicate the potential utility of the designed nano composites as drug nano sensors and initiators for destruction of the aqueous drug molecules leading to near complete mineralisations.

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CONFLICT OF INTEREST: None declared

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