Heavy metal detection in industrial waste water using Ficus Benjamina leaf extract mediated Ag nanoparticles

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Research Article

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Abstract

Polycrystalline Ag NPs were synthesized by environment benign and cost effective green route method using *Ficus Benjamina* leaf extract (FBLE). As-synthesized Ag NPs were characterized using various techniques such as X-ray diffraction, Fourier Transform Infrared spectroscopy (FTIR), Field emission scanning electron microscopy (FESEM), Transmission electron microscopy (TEM), and Atomic absorption spectroscopy (AAS). Structural analysis was carried out by employing the Rietveld refinement method which revealed that FBLE: Ag NPs exhibited face centered cubic crystallinity with space group $Fm\bar{3}m$ and space group no 225. FTIR spectra displayed the existence of phytochemicals such as phenols belonging to hydroxyl group (-OH) as bending vibration appeared at 3435 cm^{-1} and 1638 cm^{-1}, respectively. Surface morphology and microstructure of FBLE: Ag NPs were depicted using FESEM and it was observed that biosynthesized Ag NPs showed well interlinked and homogenous distribution of grains with an average grain size of 31.12 ± 0.44 nm. FBLE: Ag NPs were used to detect heavy metals such as Lead (Pb), Cadmium (Cd), and Zinc (Zn) present in industrial waste water of different factories including textile, steel, and chemical.

Highlights

- FBLE: Ag NPs possessed face centred cubic crystallinity.
- FBLE acts as capping, reducing and stabilizing agent.
- Dose dependent efficiency was observed as maximum at 90 min of exposure time to detect heavy metals.

1. Introduction

In the past few years, the novel methodology for the synthesis of metallic nanostructures with desirable morphologies and dimensions has become a challenge in the field of nanotechnology. Nanotechnology has a wide range of applications in various fields such as the pharma industry, food industry, agriculture industry, and many more (Praba et al. 2015). Metallic nanoparticles such as gold (Au), silver (Ag), and Zinc (Zn) have gained attention in the field of nano-biotechnology for the synthesis of novel nanostructures via green synthesis. Among various metallic nanoparticles Ag nanoparticles because of their well-defined optical and electronic properties in nano-regime, stands out to be a potential candidate in applications of photonics, pharmaceuticals, anti-microbial, anticancer, pollution resistant, and so on (Nayak et al. 2016; Eid et al. 2020). The toxicity of the chemicals involved in the chemical synthesis route has affected their application badly in the field of the biomedical. Therefore, eco-friendly and bio-synthesis routes are required for the synthesis of such NPs. Phytonanotechnology is a branch of nanotechnology in which we employ plant extract for the synthesis of nanostructures (Gloe et al. 2022). Green synthesis ensures maximum safety and efficiency of such NPs in their way out in the biomedical industry, food industry, or pharma industry and is also cost effective technique. In synthesis via the green route, we use eco-friendly solvents, non-toxic reagents, and green reducing agents (Mohan et al. 2014).

Various plants extract were used in literature for the synthesis of silver NPs. Among the variety of herbal plants, we opted for *Ficus benghalensis* also named as “Banyan”. It belongs to the family of “Moraceae” and also popular as “Weeping fig” or “Ficus tree”. This is a houseplant that helps in the purification of air from Xylene, toluene and other chemical products present in the atmosphere. It is commonly found tree in India, and considered as an Ayurvedic plant contains bioactive products like Vitamin A and C, alongwith flavonoids and enzymes used in the treatment of various kind of respiratory disorders alongwith skin treatment, and as an antiseptic agent too (Shukla et al. 2004; Cokuysal et al. 2006; Yarmolinsky 2012). In the synthesis of AG NPs vis green route these FICUS benzamine leaf extract act as a capping and reducing agent.

Nowadays water treatment is very important issue as industrial waste pollutes water quality very severely. The presence of organic and inorganic content in waste water is highly toxic and dangerous both for human health and environmental health. Nanoparticles synthesized via biodegradable materials such as plant extracts has reduced the rate of contamination as well as reduce the reaction time to treat the contaminated water to reduce wastage of water and environmental pollution (Goutam et al. 2020).

Martinez et al. (2016) synthesized Fe NPs using *Eucalyptus globulus* plant extract for the Arsenic absorbtion and found to be effective for the same. Qahtani et al. (2017) prepared Ag NPs from Ficus tree leaf extract for the treatment of Cadmium form the waste water of industry. It was reported that with increasing dosage of these bio synthesized Silver NPs percentage of Cd in wastewater has been reduced to a considerable amount. Aziz et al. (2019) prepared bimetallic Fe/Cu NPs using Ficus benzamine leaf extract for the removal of Carbamazepine from the aqueous solution.The removal efficiency of 95% has been achieved with the dose amount of 0.4g/L when exposed for 20 min. Ituen et al. (2021) prepared Ag NPs from Walnut extracts for treatment of Cr and Cd in petroleum wastewater and observed that synthesized NPs reduced the key water pollution indicators to great extent. 0.75g of Walnut husk extract based Ag NPs shows the maximum efficiency at room temperature for the removal of Pb, Cr, and Cd.

In the present study, Ag NPs were synthesized using Ficus benzamina leaf extract using costeffective and eco-friendly way and their structural and microstructural properties were investigated. Biosynthesized Ag NPs were used to detect heavy metals present in industrial waste water collected from textile, steel, and chemical companies.

2. Experimental

2.1 Preparation of leaves extract

In the present dissertation, *Ficus Benjamina* (FB) leaves were collected from the local garden. The leaves were washed separately with de-ionized water for few times and dried at 50°C for 4 hours. The dried leaves of all the plants were thoroughly grounded with the help of pestle mortar and then 20 g of the powder is mixed into 200 mL of distilled water and boiled at 70°C for 3 hours. After the boiling the leaves extract cooled down at room temperature. The final solution was filtered with Whatman no. 1 filter paper to separate the solid particles from the extract. The extracts obtained from the leaves was stored at 4°C for further use. The depiction of the preparation of leaf extract of Ficus Benjamina is shown in Fig. 1.

2.2 Materials

Silver nitrate [$AgNO_3$], Titanium tetra isopropoxide [$Ti\{OCH(CH_3)_2\}_4$, and Whatman no. 1 filter paper.

2.3 Synthesis of Ag NPs using *Ficus Benjamina (FB)* leaves extract

17 gm of $AgNO_3$ was dissolved in 100 mL of double distilled water (DDW) in a flask to form 1 mM aqueous solution of $AgNO_3$. 20 mL of *FB l*eaves extract was added to 100 mL of $AgNO_3$ aqueous solution. The mixture was magnetically stirred at 80°C at 900 revolutions per minute (r.p.m) for 4 hours. After stirring, Ag NPs were obtained by centrifugation at 5000 r.p.m for 30 minutes. Ag NPs were obtained after filtration using Whatman no. 1 filter paper. Ag NPs were washed with ethanol twice and dried at room temperature. As-synthesized Ag NPs were grinded using pestle mortar and fine powder was obtained for further characterization. Figure 1 illustrates the steps involved in preparation of Ag NPs using leaf extracts of *FB*.

2.4 Characterization of FBLE: Ag NPs

X-ray diffraction method was used to determine structural information of as-synthesised FBLE:Ag NPs using PANalytical X'pert Pro instrument. The phytochemical present in FBLE capped Ag NPs were detected using Fourier Transorm Infrared Spectrometer of Perkin Elmer. Surface morphology and granular microstructure of FBLE: Ag NPs was depicted using Field Emission Scanning Electron Microscope (Hitachi). The heavy metals detection was carried out using Flame Atomic Absorption Spectrometer by Perkin Elmer.

3 Results And Discussion

3.1 Structural studies

The crystal structure of FBLE mediated Ag NPs (FBLE: Ag NPs) synthesized via green route, was determined using the X-ray diffraction method. XRD diffractogram was recorded at a scanning rate of 1 °/min within a scanning angle range of 20° ≤ θ ≤ 80°. For crystallographic information, the XRD pattern was refined using the Rietveld refinement method by making use of Fullprof suit software (Sharma et al. 2021). Pseudo-voigt fitting profile with linear interpolation background was chosen to fit the diffraction pattern. XRD profile was fitted well with $Fm\bar{3}m$ space group (space group no. 227) with low goodness of fit parameter (GOF ~ 1.74) (Sharma et al. 2021). The refined XRD profile of as-synthesized FBLE mediated Ag NPs is shown in Fig. 2 All the characteristic peaks are marked with (111), (200), (220), (311), (222), and (400) planes. Few unassigned peaks are shown in Fig. 2 belonging to the organic phase present due to FBLE. XRD results are also verified with existing JCPDS data card no. 4-0787. Green route synthesized Ag NPs displayed face-centered cubic crystallinity (Khalil et al. 2014). The unit cell ball structure of Ag NPs is depicted in Fig. 3. The average crystallite size was calculated using Debye Scherrer's relation (Arockia et al. 2015):

$$D = \frac{0.9\lambda}{\beta_{hkl} cos\theta_B} \ldots\ldots\ldots (1)$$

Here, 0.9 is the shape parameter, λ is the wavelength of the X-ray source, β_{hkl} is the full-width half maxima, and θ_B is Bragg's angle. The average crystallite size was found to be ~ 18 nm. Table.1 shows the lattice parameter, cell volume, atomic position, and statistical parameter of FBLE: Ag NPs.

Table.1 The lattice parameter, atomic positions, bond length and unit cell volume of FBLE mediated Ag NPs.

Sample	Lattice Parameters (Å)	Atomic Positions				Bond length	
	a	**Atom**	**x**	**y**	**z**	(Å)	
FBLE: Ag NPs	4.082	Ag	0	0	0	Ag-Ag	4.082(3)
		Cell Volume($Å^3$)				GOF = 1.74	
		68.01					

3.2 FTIR spectra anlysis

FTIR spectroscopy was used to detect functional groups attached to as-synthesized FBLE mediated Ag NPs (FBLE: Ag NPs). FTIR spectra studies helped to ensure the chemical composition of Ag NPs. Figure 4 demonstrates the FTIR spectrum of FBLE mediated Ag NPs. The existing studies reported that phytochemicals present in the leaf extract acts as reducing, stabilization, and capping agents (Gautam et al. 2020). The metabolites present in FBLE: Ag NPs were observed at 3435 cm^{-1} and 1638 cm^{-1}, which were attributed to hydroxyl -OH and phenolic group, respectively (Bawazeer et al. 2021). A small bend located at 2908 cm^{-1} attributed to the methyl group (C-H). The presence of alkane C-H vibration was determined by the kink that existed at 2372 cm^{-1} (Singh et al. 2010). A peak positioned at 1400 cm^{-1} reflected the existence of C-C and C-N vibrations (Qahtani et al. 2017). The appearance of alcoholic and phenolic groups confirmed the existence of phytochemicals belonging to FBLE. These phytochemicals act as stabilizing and reducing agents which helped in the formation of Ag NPs.

3.3 Microstructural analysis

Surface morphology and inter-granular structure of FBLE: Ag NPs was analyzed using FESEM. FESEM image was obtained by depositing powder of Ag NPs on a sticky conducting carbon tape mounted on an aluminum disc. Figure 5(a) displays FESEM micrograph of FBLE: Ag NPs. It can be clearly visualised from the Fig. 5 that Ag NPs exhibited non uniform grain distribution with small traces of agglomeration, confirming the existence of phytochemicals of FBLE which are responsible for the biosynthesis of Ag NPs. The average grain size is calculated using statistical bar graph distribution of particles as shown in Fig. 5. From, the statistical analysis the average particle diameter was found to be 31.12 ± 0.44 nm. Gorbe et al. (2016) also reported the similar results for *Solanum muricatum* leaf extract mediated Ag NPs.

3.4 Selection of industrial waste water for treatment using FBLE: Ag NPs

In the present study, Ag NPs were synthesized by using *Ficus Benjamina* leaf extract (FBLE), were selected for removal of heavy metal ions from wastewater collected from different industries which include the steel industry, chemical industry, and textile industry located near Sirhind, Ludhiana, and Derabassi, Punjab, India. The pH and TDS level of waster water collected from various industries is give in Table.2.

Table.2 Chemical analsyis and pH value of wastewater collected from different industries.

Parameters	**pH**	**TDS**	**Concentration (ppm)**			
Samples			**Pb**	**Zn**	**Cd**	**Cr**
Sample 1 (Steel Industry)	6.82	870	0.6	4.9	ND*	1.8
Sample 2 (Steel Industry)	7.24	858	0.4	3.2	ND*	1.4
Sample 3 (Chemical Industry)	7.2	18700	0.2	0.9	0.1	1.0
Sample 4 (Textile Industry)	4.3	25600	0.04	0.07	0.16	0.9
Sample 5 (Textile Industry)	8.1	30000	3.6	2.8	0.25	1.34
Sample 6 (Textile Industry)	8.2	16000	4.31	0.4	0.14	1.5

3.5 Atomic Absorption Studies of FBLE: Ag NPs for heavy metal removal

For the studies about the effect of FBLE: Ag NPs on the removal of heavy metal from wastewater, samples of industrial effluents were collected from the discharge points of six different industries. These industries are of different sectors such as the steel industry, chemical industry, and textile industry in Punjab (Northern India).

Atomic Absorption Spectroscopy (AAS) Along with the heavy metal analysis, some other parameters were also analyzed (Table.2 and Fig. 6). The selected heavy metals (Lead, Zinc, Cadmium, and Chromium,) were analysed by Flame Atomic Absorption spectroscopy.

Based on available different concentrations of heavy metals in industrial effluents, sample 5 was selected from experimental studies. The flame atomic absorption spectroscopy (Flame AAS) method was used to study the effect of FBLE: Ag NPs on the removal of heavy metals from the solution. For this in the first step, the pH of sample 5 was decreased from 8.1 to 7.0 by adding a small amount of HCl. In the second step, a different amount (0.1/0.5/1.0 gm/L^{-1}) of FBLE: Ag NPs were added to the neutral sample solution. Then around 40 ml of solution was taken in a falcon tube and shaken at 100 rpm up to 70–80 min at 25°C. In the next step, separation of solid/liquid phase was carried out by centrifugation at 2000 r.p.m and after the filtration, estimation of the concentration of heavy metal was measured by atomic absorption spectroscopy (model: Thermo AAS 301 by Thermo Fisher Scientific).

The pH of the aqueous solution plays a significant role in the process of adsorption as pH affects the adsorbent surface (Klapiszewski et al. 2015; Hartmann et al. 2014). In this work, all studies were made with the neutral pH (original pH of the industry effluent; sample 5, was 8.1). Chamjangali et al. (2015) reported that a solution with decreasing the pH of the solution lowers the adsorption of the metal ions due to hydronation of functional groups. With decreasing the pH, H^+ ions dominate on the binding sites of FBLE: Ag NPs which also supports the hydronation of functional groups. However, it was observed that the adsorption of metal ions increases with increasing the contact time with the nano adsorbents. The effect of removal concerning contact time with increasing the dose of sorbent mass is shown in Fig. 7(a-d). It was observed that each examined metal ion was adsorbed significantly as a function of time. However, the maximum removal efficiency was observed after the 90 min of contact time without significant change up to 195 min. Figure 7(d) shows the comparative analysis of removal of different heavy metals (Pb, Zn, Cd, and Cr) with different doses of sorbents for contact time between 90–120 mins. An increment in removal efficiency was observed with the increase in the sorbent mass as well as contact time. An increment in the absorbent dosage from 0.1 to 1 gL^{-1} increases the removal efficiency of Pb, Zn, Cd, and Cr. This increment in removal efficiency of heavy metals is apparently due to the increasing number of binding sites with the increased amount of absorbent. The effect of contact time on the removal efficiency of nano-adsorbents on heavy metals was also studied. It was observed that there is no significant increment in the removal efficiency between 90–180 mins. So, a comparative study was plotted in a graph [Fig. 7(d)], which indicates that the removal percentage of different heavy metals in waste water increases with increasing the dose of FBLE mediated Ag NPs and with increasing the contact time.

Conclusion

In summary, polycrystalline FBLE:Ag NPs were synthesized via green route with cost effective and environmental benign way. Structural characterization revealed that FBLE:Ag NPs possessed face centred cubic crystal structure with small traces of organic solvent due to phytochemicals present in FBLE as confirmed by Rietveld refinement technique. FTIR spectra showed characteristics hump of phenol group positioned at 3435 cm^{-1}, which attributes to metabolites present in FBLE. It illustrates that FBLE acts as a stabilising, reducing and capping agent which participates in the formation of AG NPs. FESEM micrographs of FBLE:Ag NPs depicts non uniform distribution of grains with signature of agglomeration. The mean grain size evaluated using statistical analysis was found to be 31.12 ± 0.44nm. FBLE: Ag NPs were used to detect heavy metal impurities in industrial waste water collected from various factories such as textile, steel and chemical located at Sirhind, Derabassi, and Ludhiana, Punjab, India. Atomic absorption spectroscopic technique was used to confirm the presence of heavy metal in sample water and it was found that each heavy metal including lead, zinc, cadmium and chromium was adsorbed as a function of time. Dose dependent removal (in %) efficiency was depicted. Maximum removal efficiency was observed after 90 minutes at 1 g/L concentration of FBLE: Ag NPs. The present finding portrays that FBLE:Ag NPs showed potential candidature against industrial waste water treatment for the detection of heavy metal.

Declarations

Acknowledgment

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Compliance with ethical standards

The authors declare that the present work does not involve any human and animal involvement in the study and all the ethical standards are adapted and followed to carry out this work.

Conflict of Interest

The authors hereby declare that we have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability statement

The datasets generated during the current study will be available from the corresponding author on reasonable request.

Author’s contribution:

1. Conceptualization: Naveen Kumar

2. Supervision: Ranvir singh Panwar

3. Methodology: Priyanka Singh and Nupur Aggarwal

4. Analysis: Harvinder Kaur Sidhu, Gagan Anand and Navdeep Sharma

5. Proof reading: Srinu Nagireddi

6. Synthesis: Saransh Malhotra

7. Manuscript drafting: Anjana Sharma

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Consent to Publish

I, Ranvir Singh Panwar, on behalf of all the authors give my consent for the publication of present report in your esteemed journal.

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Figures

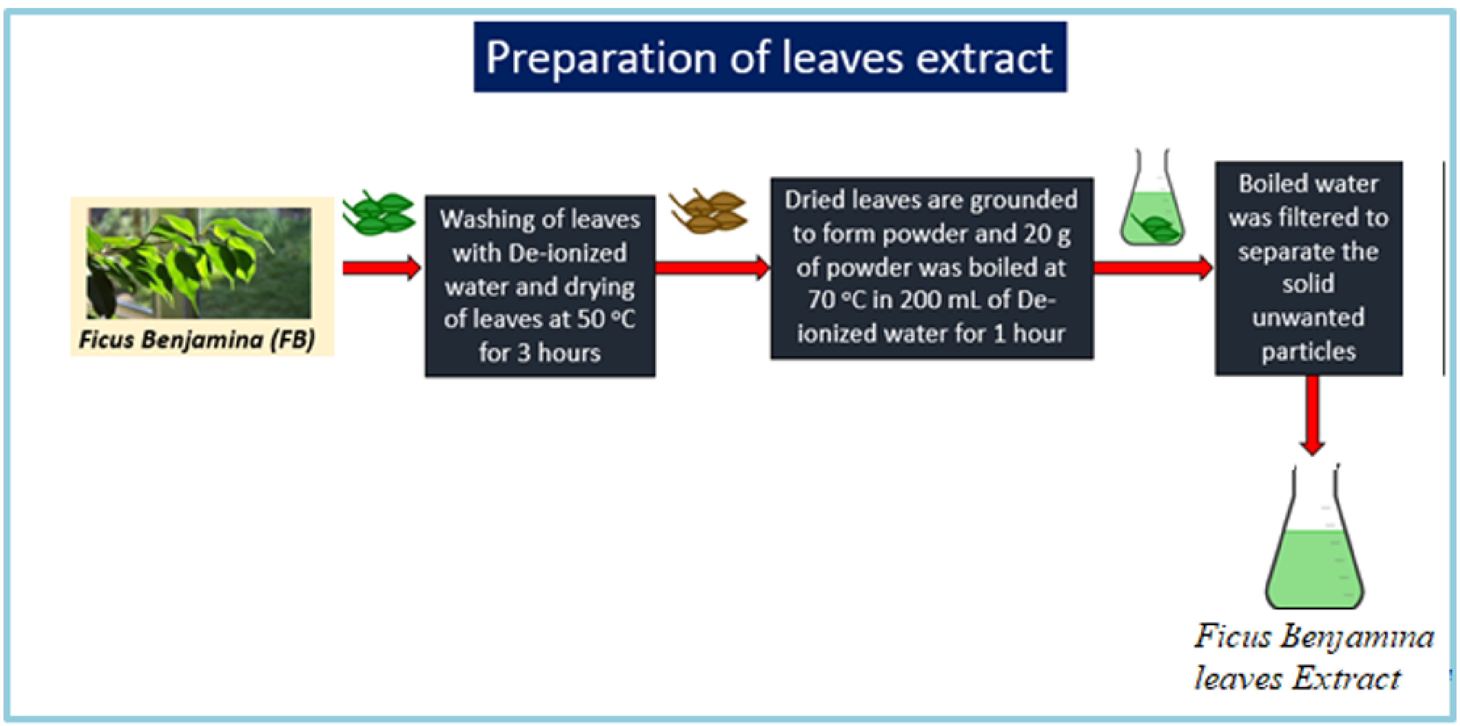

Figure 1

Schematic depiction of preparation of leaf extract of *Ficus Benjamina*.

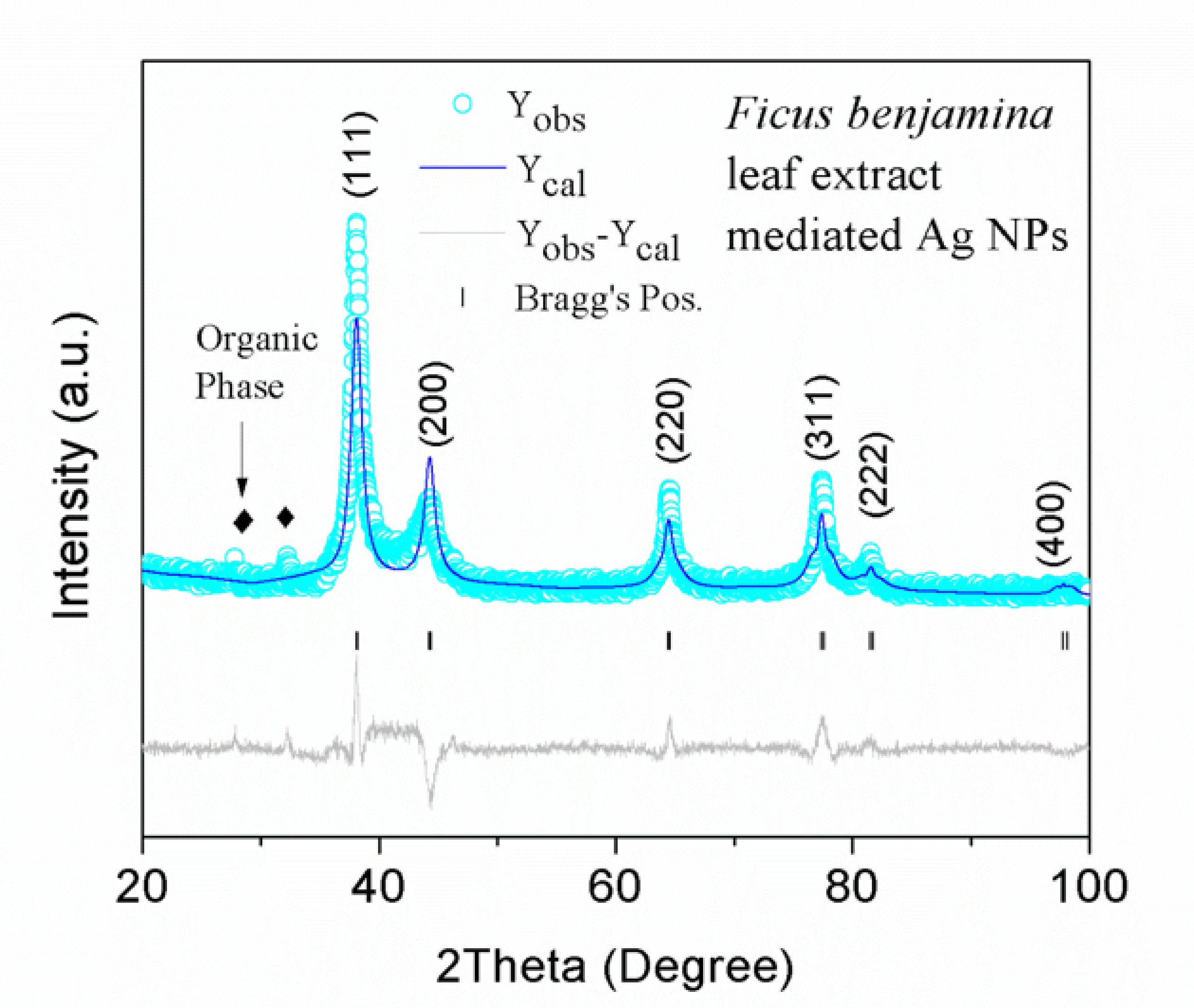

Figure 2

XRD pattern of FBLE mediated Ag NPs.

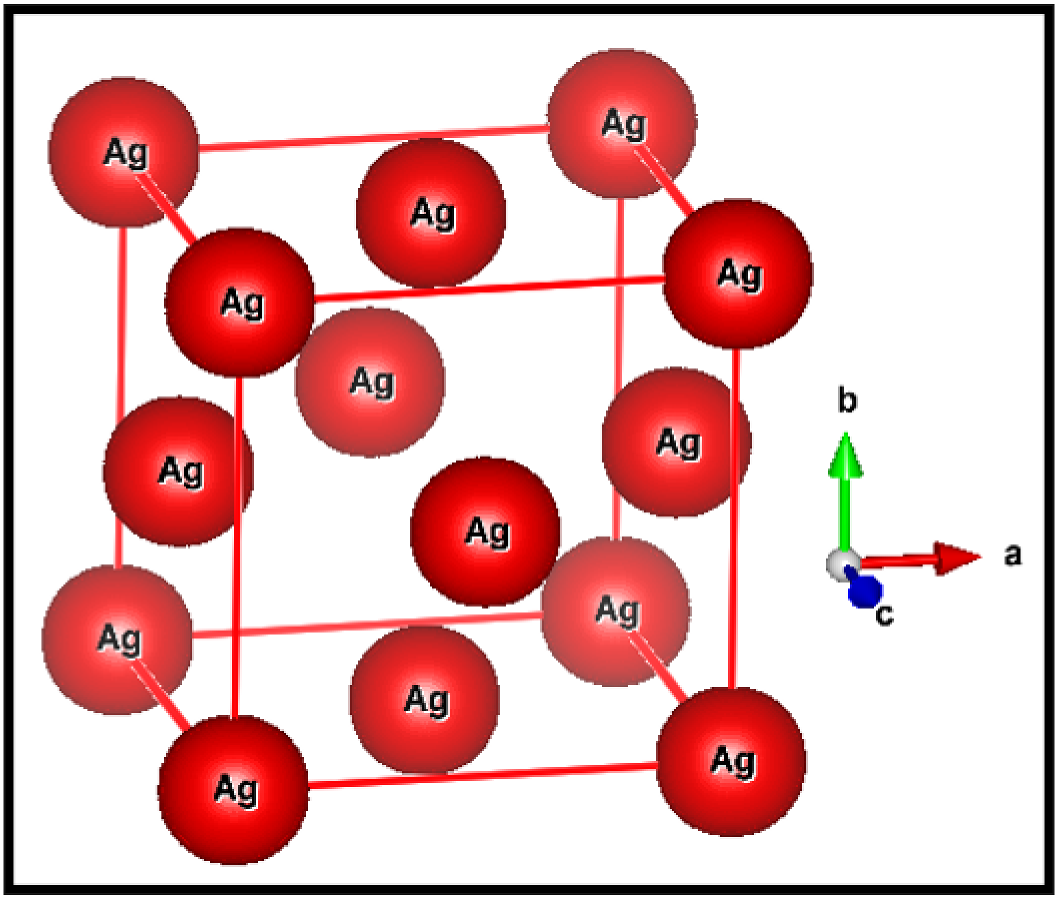

Figure 3

Ball structure of FBLE mediated Ag NPs illustrating face-centered cubic crystallinity.

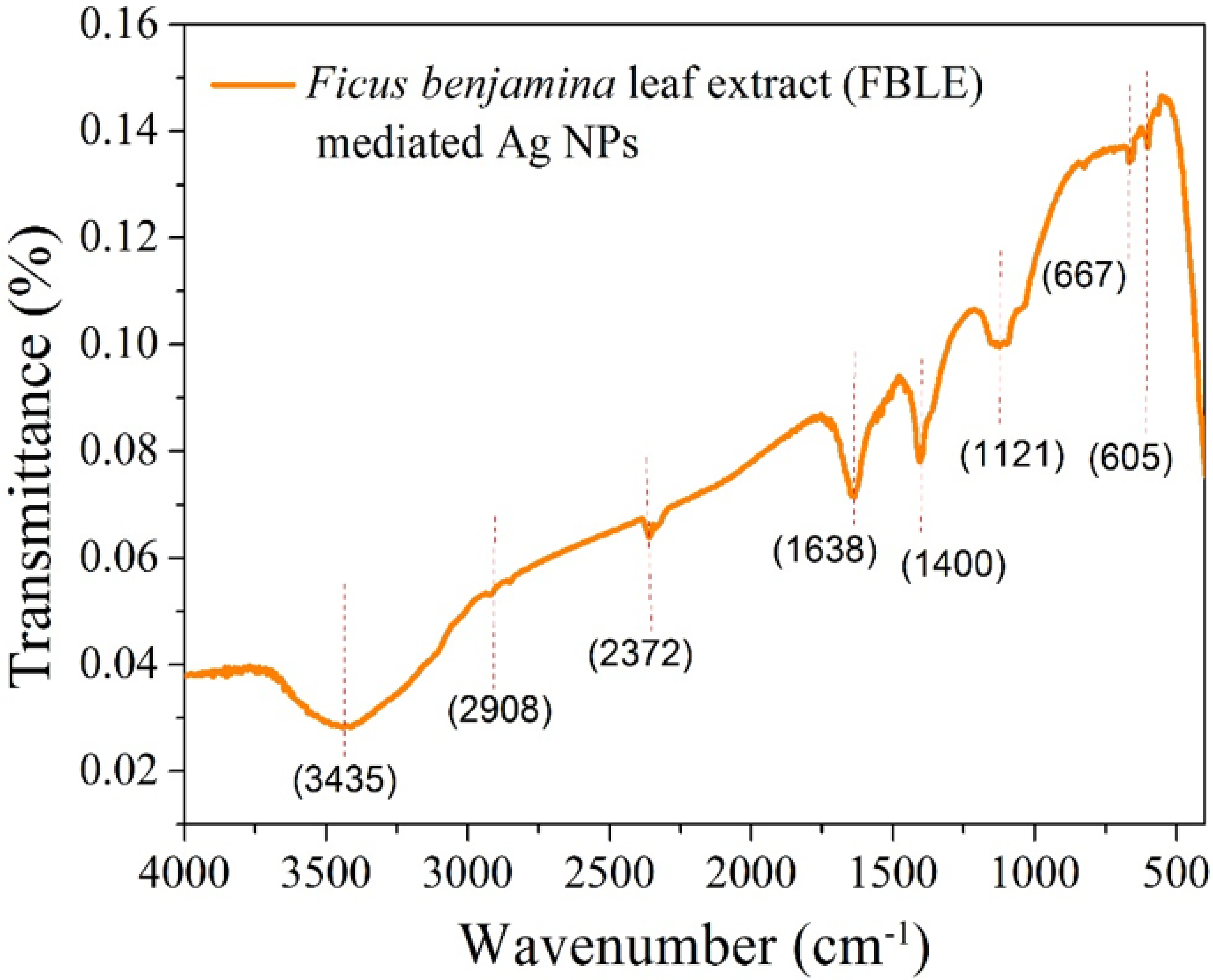

Figure 4

FTIR spectrum of FBLE mediated Ag NPs.

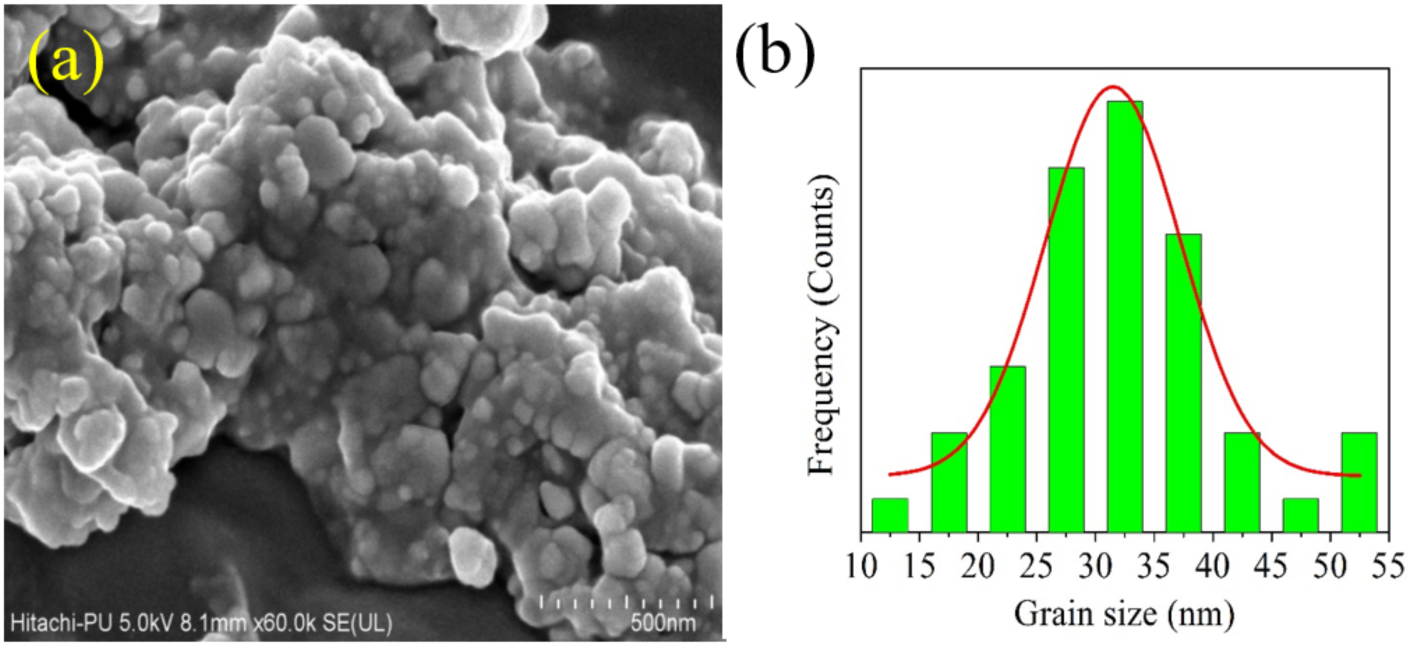

Figure 5

(a) FESEM image of FBLE mediated Ag NPs and (b) Histogram depicting bar graph distribution of grains.

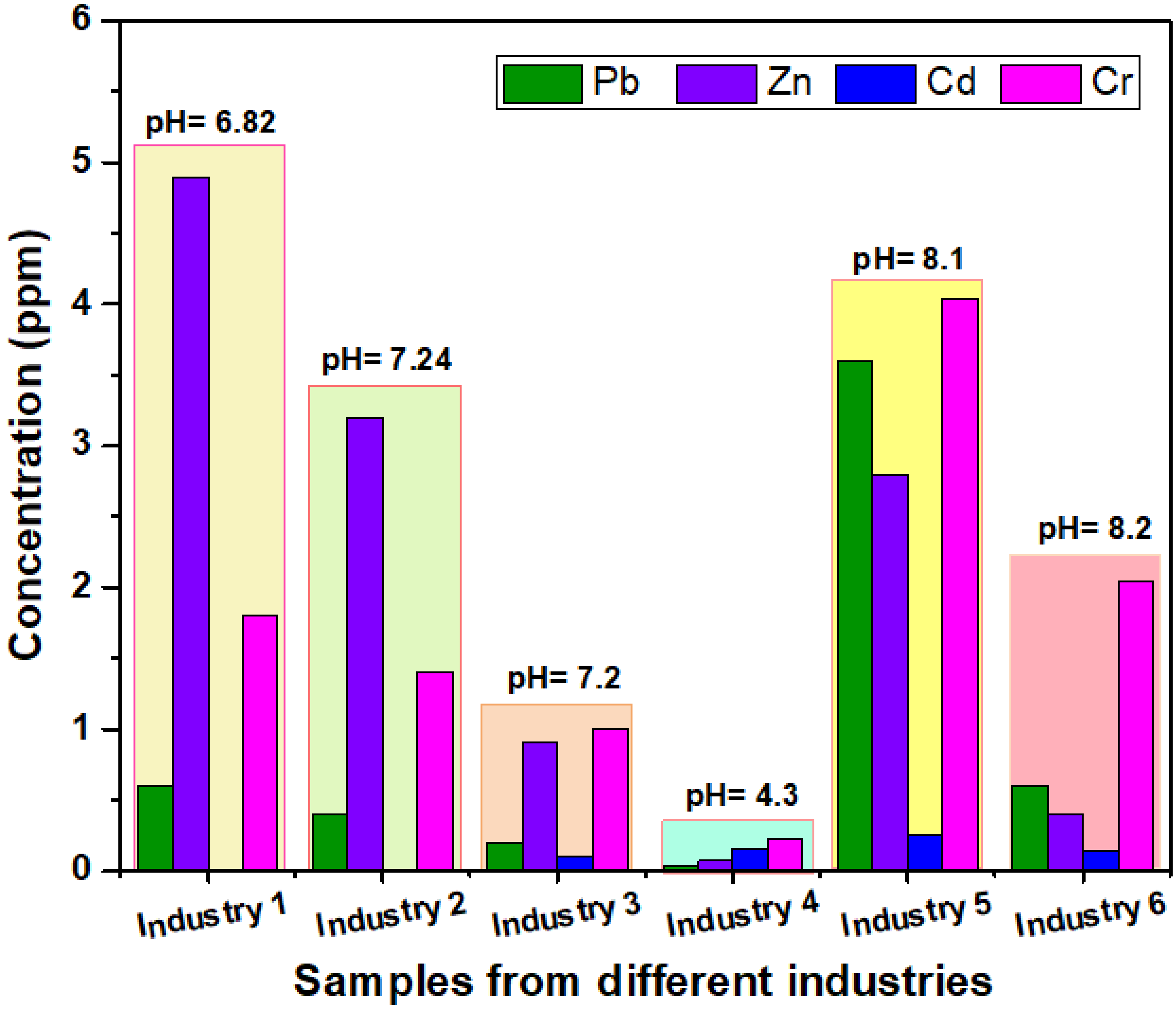

Figure 6

Concentration of heavy metals and pH of the different samples.

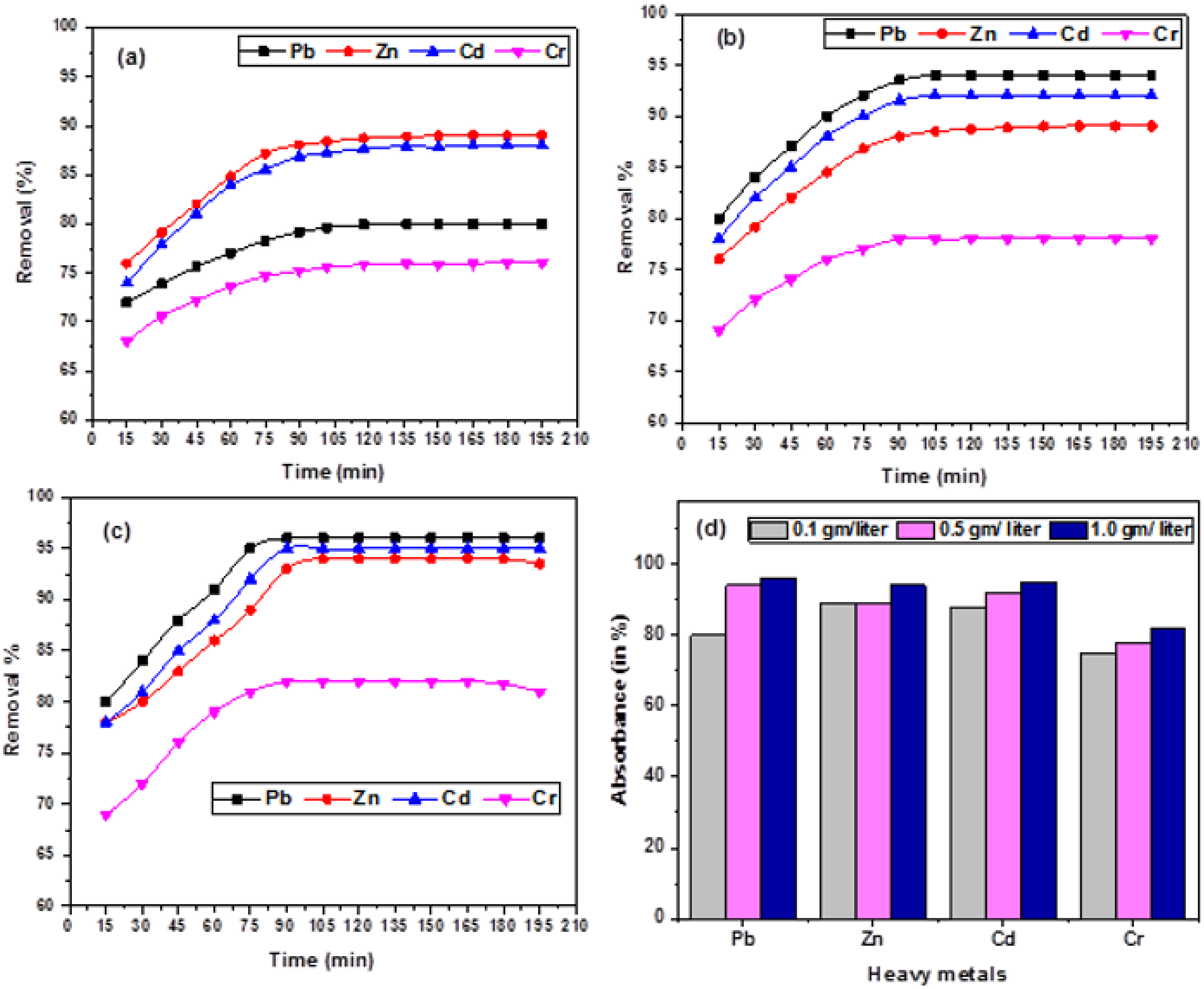

Figure 7

Effect of contact time on adsorption of heavy metals onto different adsorbents doses (a) 0.1 gmL^{-1}, (b) 0.5 gmL^{-1}, (c) 1.0 gmL^{-1} and (d) Comparative analysis of absorbance for contact time between 90- 120 mins.