



## Original Research Article

# Allium based green route synthesized silver nanoparticles for removal of polycyclic aromatic hydrocarbons

Manish S. Sengar<sup>a,b</sup>, Sachin Saxena<sup>a,b,\*</sup> , Anita Lakhani<sup>a</sup>, Soami P. Satsangee<sup>b</sup>

<sup>a</sup> Department of Chemistry, Dayalbagh Educational Institute, Dayalbagh, Agra-282005, India

<sup>b</sup> USIC, Dayalbagh Educational Institute, Dayalbagh, Agra-282005, India

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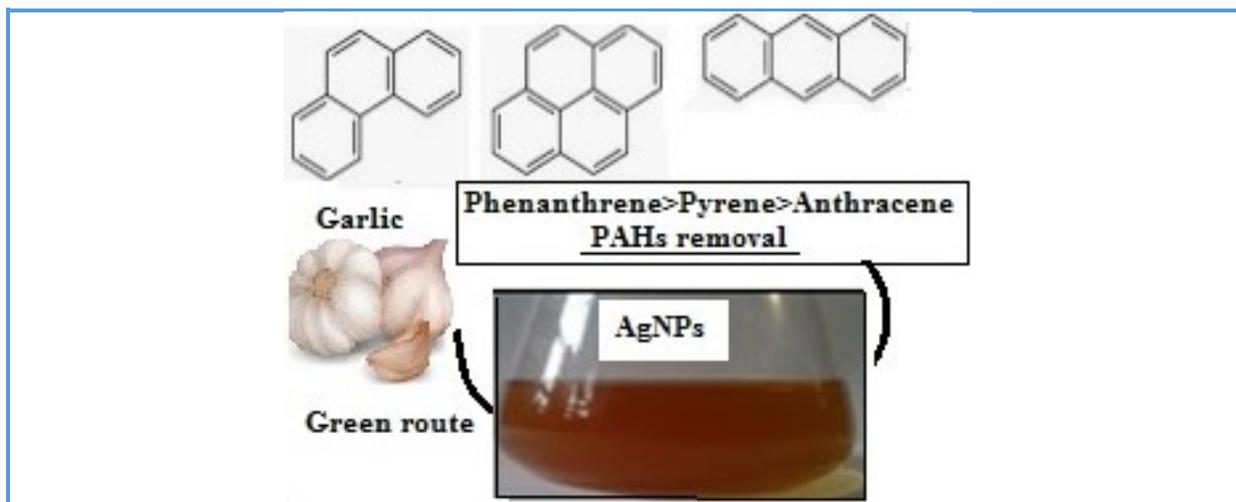
Garlic extract etc.

## ABSTRACT

This paper presents removal of some polycyclic aromatic hydrocarbons (PAHs) by silver nanoparticles (AgNPs) which were synthesised by garlic based green route method. *Allivum sativum* (Garlic) extract was used for the synthesis of silver nanoparticles Ag(P) as a green route process while synthetic chemical method was adopted for preparation of silver nanoparticles Ag(W). The synthesis of AgNPs was studied as a function of variation in volume of garlic extract, temperature and time. Solution of pyrene, anthracene and phenanthrene was prepared in n-hexane, and their removal efficiencies were studied. PAHs were removed successfully with optimal efficiency of more than 80% with the affinity order of removal, which followed Phenanthrene > Pyrene > Anthracene. The adsorption of PAHs on AgNPs is attributed to the occurrence of hydrophobic interactions. Ag (P) nanoparticles synthesized were found relatively better adsorbent than Ag (W), for removal of PAHs. It may be ascribed owing to the presence of more functional groups in the garlic extract participating in binding of PAH to the surface. The adsorption property of the AgNPs synthesized by both ways was studied and comparative results were obtained based on adsorption efficiency of PAHs. Further, FTIR and XRD were used to characterize the properties of garlic extract with binding and interaction taking place between AgNPs and PAHs. UV-Vis confirmed the formation of Ag(P) and Ag(W) by biological method and chemical wet co-precipitation method, respectively. Ag(P) can be applied over a wide range of temperature, due to stability of compounds present in *Allivum sativum* at high temperatures.

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## Graphical Abstract



## Introduction

Regarding PAHs (polycyclic aromatic hydrocarbons) there is more than two aromatic rings form the basic skeleton of these type of compounds. These substances with their oxy- and nitro-derivatives, occur as complex mixtures in all environmental compartments [1]. Both anthropogenic and natural sources are responsible for the PAHs in an environment. The common PAH natural sources include volcanic eruptions, effusion of trees, forest fires, and oil seepage. Relatively, anthropogenic sources are responsible for the presence of huge percentages of several PAHs in the surroundings, which mainly includes wood burning, municipal and industrial waste incineration, coke, asphalt roads, roofing tar, combustion of fossil fuels, coal tar, discharges from industrial plants, coal gasification sites, waste water treatment plants, smoke houses, cigarette smoke, atmospheric contamination of leafy plants, and hazardous waste sites. PAHs are formed mainly through pyrolytic process, where combustion of organic materials occur in an incomplete manner during human and other industrial activities, such as crude oil

processing and coal, refuse combustion, and natural processes such as carbonization [2]. Some PAHs with 3, 4 or more benzene rings, such as benzo [a] anthracene, chrysene and benzo [a] pyrene, can generate covalent DNA adducts and oxidative DNA lesions, resulting in mutagenic and carcinogenic effects [3]. Moreover, prolonged adverse effects of persistent PAHs are observed in the environment [4]. U.S. Environmental Protection Agency (US EPA) claimed 16 PAH as primary pollutants to the environment. Though, transportation and logistics have exponentially increased the levels of PAHs at an alarming rate [5, 6]. Recently, accidents have been reported with spillage of million barrels of oil containing huge amounts of PAHs into water bodies, e.g., Port of Dalian in China [7] causing prolonged threatening to the marine ecosystem. Therefore, there is an urgent need to research and effective technology for the removal of PAHs from environment [8, 9].

Kow greater than 1000 is an indicator of hydrophobic behaviour of PAHs, which increases with increase in aromatic rings. An inverse relationship is observed between the molecular weight and volatility of PAHs. Similar

is the relationship with Henry constant and molecular weight of the compounds. Therefore, with an increase in molecular weight of the compounds, volatility and Henry constant decreases [10, 11]. Various approaches have been made to reduce the costs in order to treat contaminated areas and remove these toxic aromatic compounds [12]. Traditional techniques found in literature includes processes such as bio-degradation [13], ozonisation [14], electro-oxidation [15] and photo catalysis [16] which are observed to be insufficient methods for the removal of PAHs. Open waters with in situ removal of PAHs demands a feasible method i.e. Sorption treatment [17, 18].

Literature is enriched with traditional sorbents which have ability towards remediation and removal of toxic substances. Though low adsorption efficiencies and capacities of these sorbents limit their applicability. This aspect is effectively removed by nanoparticles that exhibit high specific surface area with much faster rates of interaction and higher efficiency in treating waste water [19]. Pollutants (inorganic and organic) removal has been effectively observed in case of nanoadsorbents with high adsorption capacity and interaction ability [20]. Nowadays, metallic nanoparticles have been extensively used for fabrication of different electrochemical and electronic sensors based on specific size-dependent properties of nanoparticles [21-23]. Further, green synthesis of Ag nanoparticles has been reported with use of different solution systems, surfactants [24] and strong reductants (e.g., borohydride or hydrazine) [25, 26], which is generating hazardous waste in enormous amounts. Hence, greener protocols and methodologies are needed utmost, which can minimize the use of toxic and hazardous reagents [27] that pursue and promote cost-effective alternatives, and adoption of

numerous nanoparticles applications. Phytochemicals are good stabilizing and reducing agents which naturally exhibits high phytopotentials, which is helpful in synthesizing green nanoparticles at ambient temperature. For example, Lee et al. have shown the synthesis of non-toxic gold nanoparticles from honey (diameter ~15 nm) and soyabean extract, respectively [28]. Their stability in biological media and enhanced therapeutic in-vivo activities provide the reason for researchers and medical experts to rely on naturally synthesized nanomaterials with minimum side effects. Recent research work on gold and silver nanoparticle synthesis have employed herbal extracts from alfalfa [29], lemongrass [30], and green tea [31], where the natural extracts serve as both reducing and stabilizing agents. Garlic extract also reports the presence of phytopotential groups, which are helpful in preparation of AgNPs [32]. The present paper aimed to develop cost effective green approach towards removal of PAHs using *Allium sativum* extract. Synthetic method was also used for the preparation of Ag(W). Further, Ag(P) prepared from garlic extract exhibited better removal efficiency as compared to Ag(W).

## Experimental

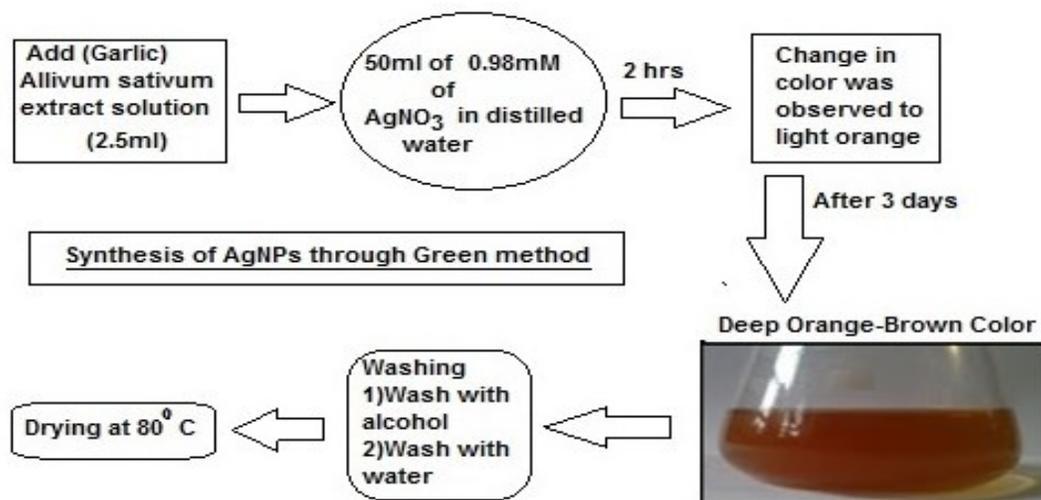
### *Materials and methods*

Silver nitrate and sodium hydroxide was purchased from Fischer scientific. ELGA purification system (U.K) was utilized for obtaining ultra-pure Milli-Q water. Cary 630 Spectrometer was used for acquiring ATR-FTIR spectra while X-Ray diffraction pattern was procured from Bruker, D-8 Advance, 2008. Shimadzu UV-Vis was used for UV-Vis spectra.

Garlic was rinsed with deionised water before use. Known weight of (approximately 5-6 g) of *Allium* were cut into pieces and added to

50 mL of deionised water, and kept at room temperature for 24 hours. The extract obtained was then used for AgNPs synthesis as shown in Figure 1. The solution formed was decanted to collect the garlic extract solution thereby removing the solid garlic pieces were removed. Ag(P) were synthesized by adding freshly prepared extract of *Allium sativum* (Garlic) in 0.98 mM silver nitrate dissolved in distilled water, after 2 hours a light orange colour appeared in the vessel, this is the first sign of

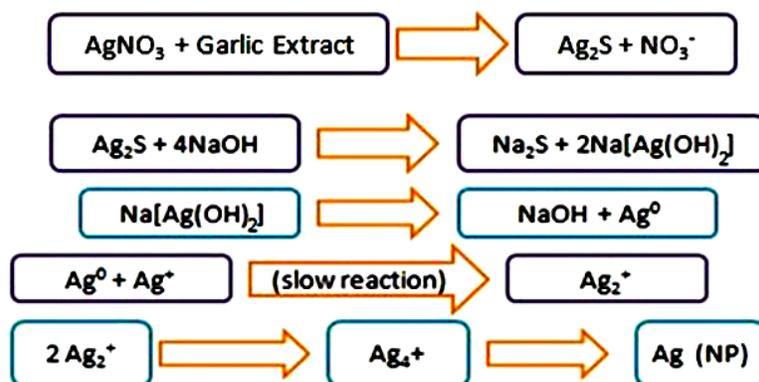
AgNPs preparation process after which it was kept at ambient temperature for 3 days, the change in deep orange brown colour confirmed the presence of nanoparticles in the solution system, this colour appeared because of AgNPs formation took place in the aqueous solution. Further, the solution was filtered and washed properly with alcohol followed by water till it becomes neutral in nature. Ag(P) was dried at 80 °C [33].



**Figure 1.** Flowchart of synthesis of silver nanoparticles

The syntheses of AgNPs were studied as a function of variation in volume of garlic extract, temperature and variation in time. Various volumes (1, 2, 3, 4, and 5 mL) of garlic extract were added to 50 mL of 1.0 mM of AgNO<sub>3</sub> in distilled water to study the effect of volume of garlic extract. The synthesis was carried out at two temperatures (25 °C and 40 °C) to study the effect of temperature. After two hours, light orange colour was observed. The solution was left to age for 7 days leading to formation of deep orange-brown colour which was analysed using UV-Visible spectrometer (full scan range 190-1100 cm<sup>-1</sup>) to confirm formation of AgNPs. Further, 2M NaOH solution (10 mL) was added to form precipitate and obtain greyish AgNPs

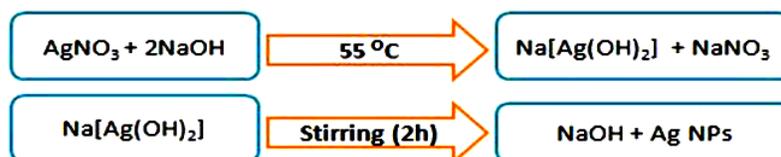
when dried under vacuum. Garlic extract led to formation of silver sulphide from silver nitrate. This silver sulphide reacts with NaOH and forms Na[Ag(OH)<sub>2</sub>] which again decomposes to sodium hydroxide and precipitates silver which has very small size as compared to reactant. Chemical reaction involved in the formation of AgNPs using garlic extract and silver nitrate solution can be seen in Figure 2. This process was completed in 3 to 5 days. It depends on the concentration of garlic extract, added into the silver nitrate solution. This formation process depends on the concentration of garlic extract but is independent of concentration of silver nitrate solution [34, 35].



**Figure 2.** Reactions occurring during synthesis of Ag (P) by green route process

AgNPs were also synthesized by wet chemical co-precipitation method. A known volume (mL) of 0.9 M aqueous solution of sodium hydroxide was heated at 55 °C followed by addition of 0.45 M aqueous solution of silver nitrate under speed stirring. The precipitate of

Ag nanoparticles was formed. These particles were cleaned with distilled water after the solution is left undisturbed for 2 h, finally air drying was done at 60 °C [36]. The mechanism occurring is shown in Figure 3.



**Figure 3.** Reactions occurring during synthesis of Ag (W) by wet chemical co-precipitation method

The synthesized AgNPs [Ag(P) and Ag(W)] were applied as adsorbents in experiments which are time based in order to analyse the removal of polyaromatic hydrocarbons. A 10 ppm of solution of pyrene, anthracene and phenanthrene was prepared in n-hexane. To assess the absorption efficiency of the synthesized nanoparticles, a known volume (2 mL) of synthesized AgNPs was added in test tubes containing 25 mL of the prepared PAHs solution at concentrations ranging between 0.1-5 ppm. The solution was stirred for 10 min on a stirrer, an aliquot was drawn after 20 min, and was further analyzed on UV-visible spectrophotometer and their absorbance were recorded at wavelengths of 241, 250 and 260

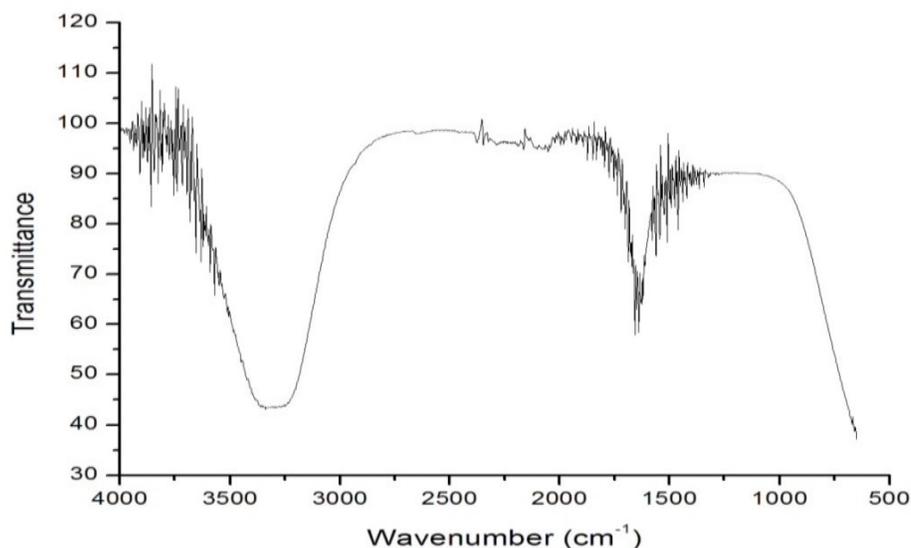
nm for pyrene, phenanthrene and anthracene respectively. The absorbance of the PAHs solution was also recorded prior to the addition of AgNPs. The amount of PAHs adsorbed (removed) was determined from the difference of the absorbance before and after addition of silver particles [32, 37].

## Results and Discussion

The FTIR spectrum of garlic extract is demonstrated in Figure 4. The different peaks observed in the spectrum and their assignment to characteristic groups is listed in Table 1. In general, the presence of C-O stretching is observed in the range 1300-1450  $\text{cm}^{-1}$ . A peak is observed at 1436  $\text{cm}^{-1}$  this may be due to esters,

carboxylic acid and alcohol. CO group stretching at  $1772.64\text{ cm}^{-1}$  is attributed to the presence of aldehyde, ketones and carboxylic acid, while the peak at  $1661.82\text{ cm}^{-1}$  arises due to presence of sulphur group in garlic extract. The intense peak in the range of  $1550 - 1650\text{ cm}^{-1}$  is due to

carboxylic group and NH bending [38, 39]. These functional groups in the extract are responsible for better adsorption phenomenon occurrence, thereby enhancing the ability to remove PAHs.



**Figure 4.** FTIR spectrum of garlic extract showing presence of different functional groups

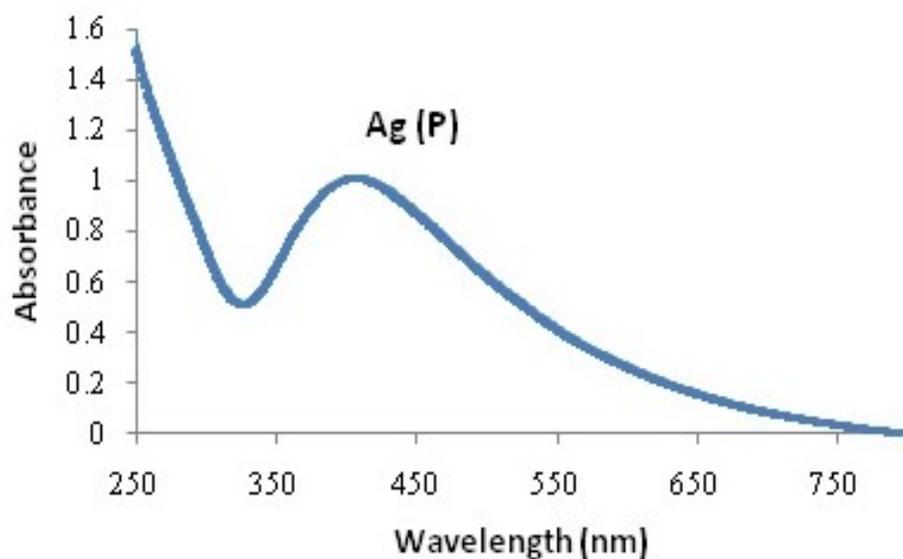
**Table 1.** Characteristics of the FTIR Spectrum of *Allium sativum*

Wavelength range ( $\text{cm}^{-1}$ )	Assignment	Peaks( $\text{cm}^{-1}$ )
3500 - 3000	Bonded -OH group	3421
2900 - 2800	CH stretching	2900
1740 - 1680	C=O groups	1748
1670 - 1640	Carboxylic group	1653
1600 - 1500	NH bending	1577
1280 - 1240	-SO <sub>3</sub> <sup>2-</sup> stretching	1260
1450 - 1300	CO stretch	1436

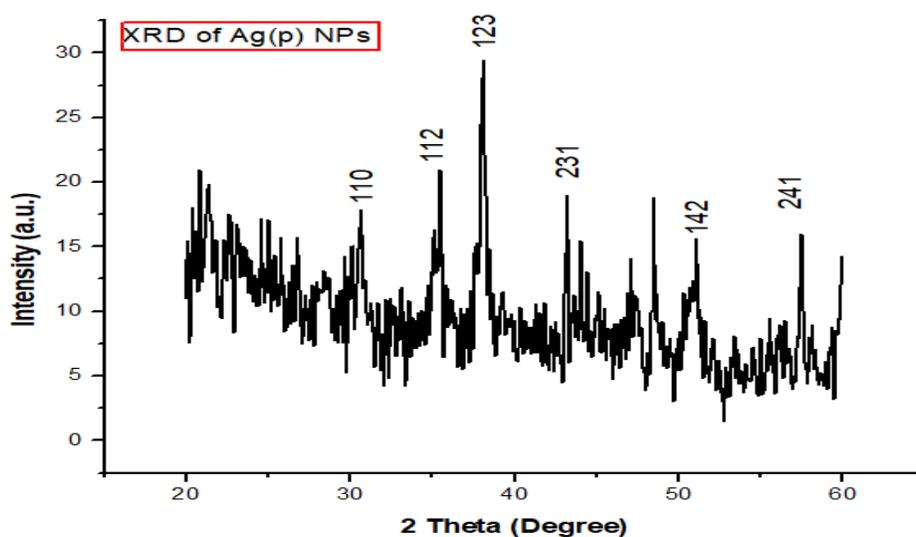
The UV-Visible spectra of prepared AgNPs are revealed in Figure 5. The absorbance can be observed in the region of 350-500 nm with characteristic peak of silver nanoparticles. The reddish-brown color of the nanoparticles was reported due to surface plasmon resonance (SPR) occurrence in the range. On adding garlic extract in AgNO<sub>3</sub> solution, the biomolecules get stabilized in the solution system by interacting with each other and with silver salt. Initiation of the process of capping, reduction and nucleation leading to formation of

nanoparticles. Similar observations have also been reported earlier [40, 41].

XRD spectrum of Ag(P) revealed diffraction peaks with 2 theta values of  $30^{\circ}57$ ,  $35^{\circ}46$ ,  $38^{\circ}12$ ,  $43^{\circ}12$ ,  $51^{\circ}09$ ,  $57^{\circ}55$  which corresponds to 110, 112, 123, 231, 142 and 241 lattice planes of AgNPs, respectively. These peaks observed at different planes is due to the presence of garlic extract that is acting as a reducing as well as capping agent during the formation of AgNPs (Figure 6).



**Figure 5.** UV-Visible spectra of AgNPs

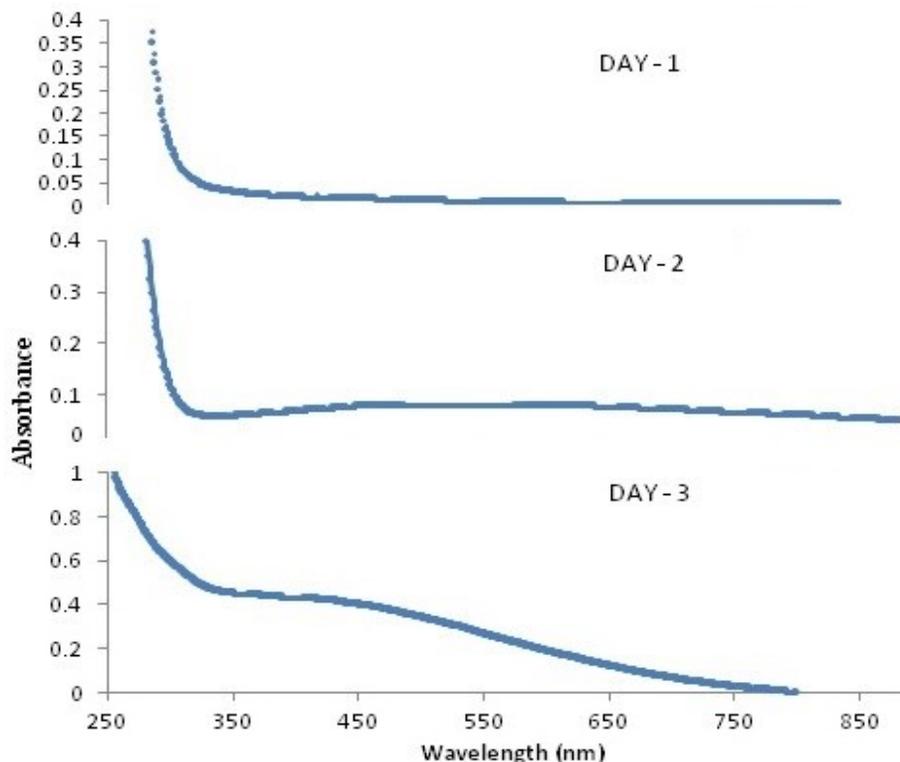


**Figure 6.** XRD of AgNPs synthesized from garlic extract Ag(P)

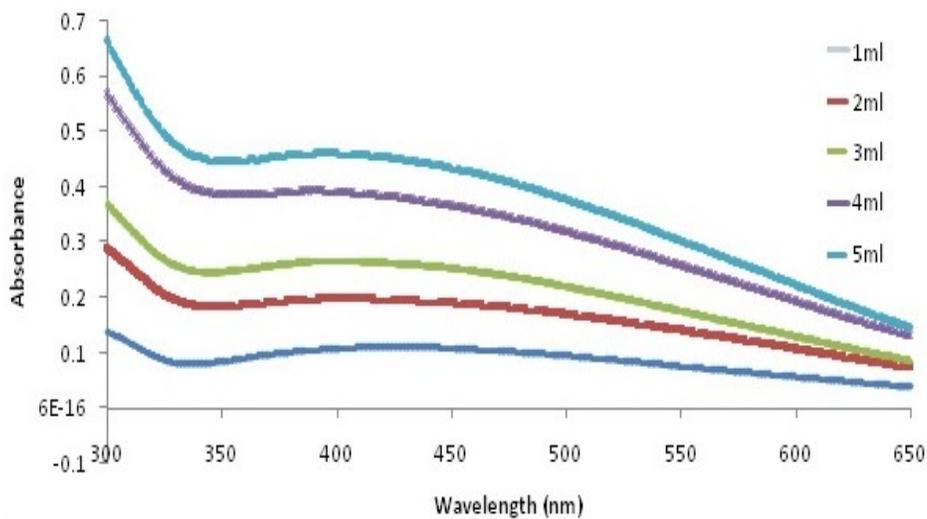
A speedy conversion of silver nitrate into AgNPs was indicated by colour changes from colorless to yellowish brown within few minutes of addition of sodium hydroxide in  $\text{AgNO}_3$  solution for preparation of AgNPs by chemical reduction method. However, in the biological method (green process) of synthesis the formation of nanoparticles was delayed as is evident from Figure 7 (day 1, 2 and 3) which

shows that the peak around 400 nm, which is due to surface plasmon resonance observed at the third day [42].

Formation of AgNPs also depends on the volume and hence the concentration of garlic extract added as is evident from Figure 8. The intensity of the absorbance observed around 400 nm increases with increase in the volume of the extract.



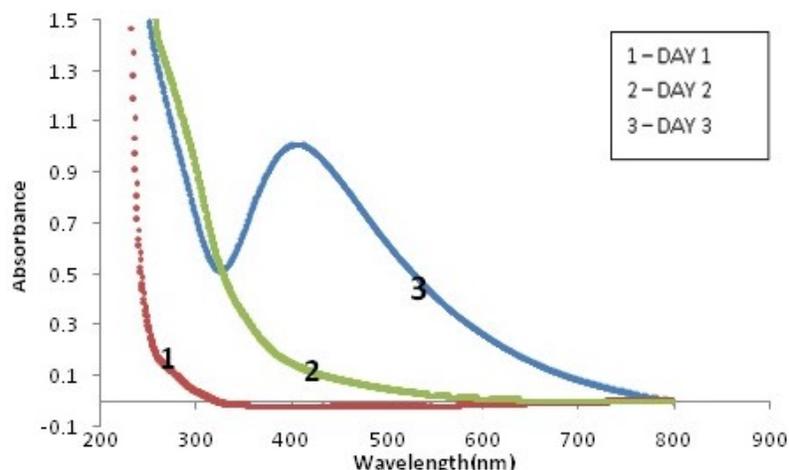
**Figure 7.** UV-Vis spectra of silver nanoparticles recorded on day 1, 2 and 3 at 25 °C



**Figure 8.** UV-Vis. spectra of AgNPs after different volumes of garlic extract used

AgNPs were formed by garlic extract at 25 °C (Figure 5) and 40 °C to study the effect of temperature on synthesis and the UV-Visible spectra were recorded on the 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> day. The UV-visible spectra as a function of

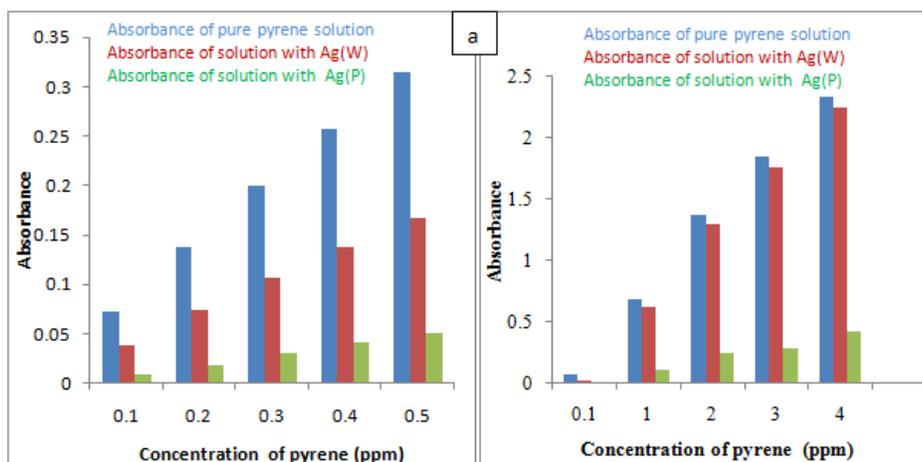
temperature and time are shown in Figure 9. The increase in the intensity of the absorbance peak at 400 nm revealed that the synthesis is also temperature dependent with aging and it increases with increase in temperature.

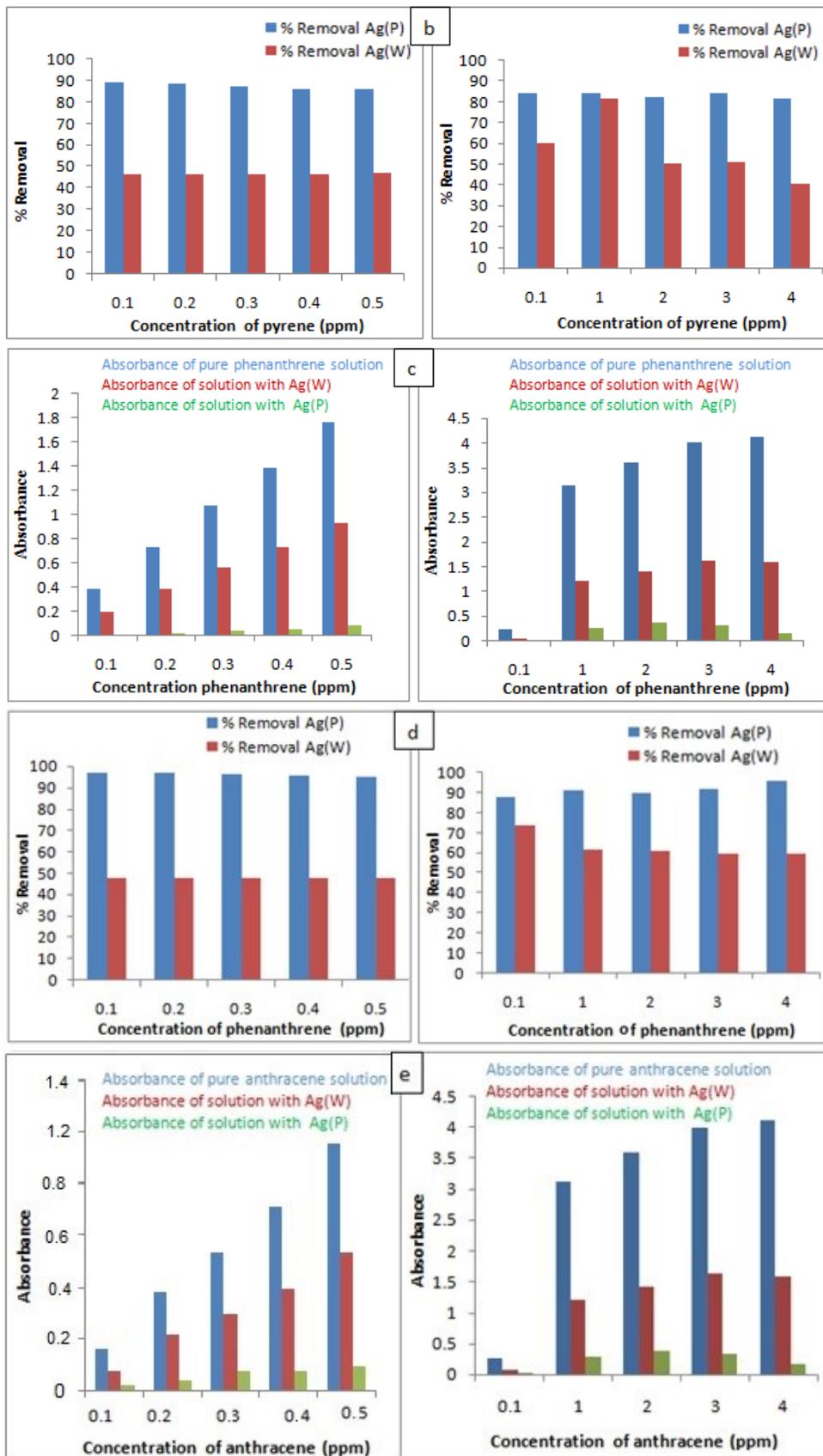


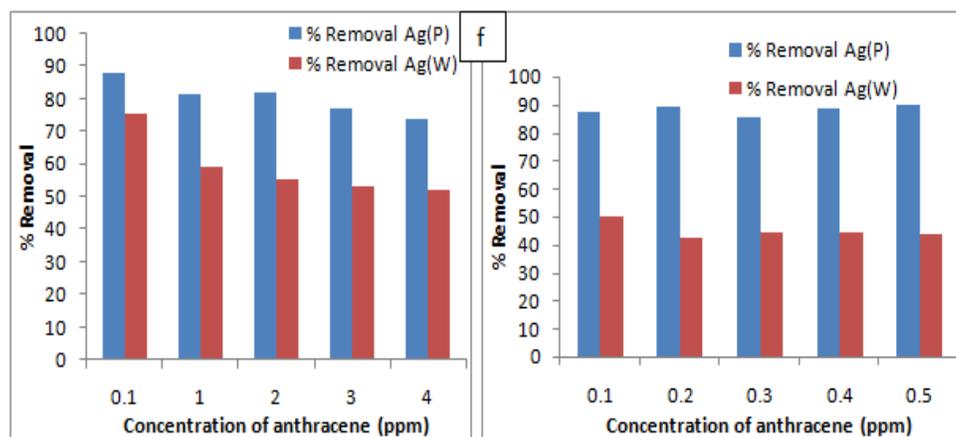
**Figure 9.** Ag (P) formation using garlic extract at a temperature 40 °C

The synthesized AgNPs [(Ag(W) and Ag(P)] were applied as adsorbents to find the removal of some selected poly aromatic hydrocarbons. The absorbance and % removal of polyaromatic hydrocarbon solution both with and without AgNPs for each hydrocarbon is depicted in [Figure 10a-f](#). It is clearly evident that both types of silver particles are good adsorbent for the removal of PAHs. At low concentrations of applied PAHs the % removal with biologically synthesized silver particles for phenanthrene ranges between 95-97%, 85-89% for anthracene and 83-89% for pyrene. At high concentrations of applied PAH the % removal for phenanthrene ranges between 87-95%, 73-87% in case of anthracene and 81-84% in case of pyrene. With chemically synthesized silver

particles, at low concentrations of applied PAHs the % removal ranged between 45-47% in case of pyrene, 42-50% in case of anthracene and 46-47% of phenanthrene. At high concentrations of applied PAHs the % removal was 40-60% in case of pyrene, 52-75% in case of anthracene and between 59-73% for phenanthrene. The effective adsorptive behavior signifies the presence of hydrophobicity of non-polar groups present in the aromatic rings [43, 44]. Kumar *et al.* [45] in 2009 has reported about the better removal of phenanthrene followed by anthracene and pyrene in the batch experiments reporting lower removal percentages of pyrene to its recalcitrant toxicity and higher molecular weight.







**Figure 10.** Bar graphs show (a, c and e) absorbance of PAHs solutions (pyrene, phenanthrene and anthracene, respectively) at different concentrations before and after treating with AgNPs and (b, d and f) percentage removal of pyrene, phenanthrene and anthracene with AgNPs [Ag(W) and Ag(P)] at different concentrations of PAHs solutions

## Conclusions

This research study offered an alternative cost effective green approach towards the removal of some toxic PAHs. *Allium sativum* extract synthesized AgNPs [Ag(P)], was found to remove PAHs successfully with more than 80% efficiency following the order of Phenanthrene>Pyrene>Anthracene. It may be described on the basis hydrophobicity aspect and presence of multiple functional groups in the plant extract participating in binding of PAH to the surface. Ag(W) synthesized by wet chemical method also removed PAHs but relatively with less efficiency as compared to Ag(P). At low concentrations of applied PAHs, removal percentage with Ag (P) and Ag (W) ranges between 95-97% & 46-47%, 85-89% & 42-50%, 83-89% & 45-47% for phenanthrene, anthracene and pyrene respectively. Further, Ag(P) can be applied over a wide range of temperature, due to stability of compounds present in *Allium sativum* at high temperatures. The study recommends the use of alternate economical and green natural adsorbents for control of PAHs.

## Disclosure Statement

No potential conflict of interest was reported by the authors.

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## Orcid

Sachin Saxena  0000-0001-9700-6676

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