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Green Synthesis and Characterisation of Copper (I) Iodide nanoparticles using kidney bean seed extract and its anti-bacterial activity

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Abstract— Copper (I) iodide is highly versatile material having applications in solar cells, semiconductors, catalysis and anti-bacterial and anti-fungal activity. The present study involves the synthesis of copperiodide nanoparticles from the kidney bean seed extract that is rich in anthocyanin content. The synthesized copper iodide was characterized using X-ray powder diffraction and SEM. The size of the CuI from X-ray powder diffraction and SEM was observed to be in the nano range. It was found that copper iodide precipitated as nano flowers. The synthesized CuI was found to show good antibacterial activity.

Index Terms— Anti-bacterial studies , copper iodide, green synthesis, nanoparticles, SEM, X-ray powder diffraction, Anthocyanin

1 Introduction

Copper iodide nanoparticles show application in several fields like catalysis, separation techniques, photonics, anti-microbial activity, drug delivery systems, etc. [1, 2]. Copper is a polymorph consisting of three crystalline phases: α, β and γ [3, 4]. .The γ -CuI has potential applications in light emitting diodes due to a large exciton binding energy which causes a high intensity of violet emission [5-7]. Literature survey shows synthesis of copper iodide by several different methods, such as iodination of Cu films, electrodeposition[8] pulse laser deposition technique[9] liquid phase reaction[10] and vacuum evaporation [11] ,ethanol thermal method [12], electrochemical/chemical method[13], coprecipitation, sol-gel processing, microemulsion, hydrothermal methods, solvo-thermal methods, template syntheses, biomimetic syntheses[14].

Although Copper (I) iodide nanoparticles synthesized via chemical route showed good dispersion in the suspension, it resulted in formation of an impure product. Therefore, there was a need for a method that employs less chemicals and gives high purity product. Synthesis of copper iodide nanoparticles using green chemistry principles is environment friendly, non-toxic and uses of less chemicals. Extracts from plants [2] may act as both reducing and capping agents in nanoparticle synthesis.

The present study aims at the synthesis of copper iodide nanoparticles using the kidney bean seed extract that is rich in anthocyanin content.

2 Materials and Methods:

2.1 Preparation of Kidney bean seed extract:

Black kidney bean seeds was washed thoroughly many times under running water and then rinsed with distilled water. The cleaned seeds were then soaked in distilled water for 24 hours. Then the soaked kidney bean seeds were ground using a mixer and made into a paste.

The resultant paste was diluted with distilled water. To get a uniform solution, the kidney bean seed solution was stirred for 3 Hrs. Subsequent solution was then centrifuged. The centrifugate was used for synthesis of Copper iodide nanoparticles.

2.2 Synthesis of Copper (I) Iodide Nanoparticles:

Cu(NO3)2.3H2O and NaI were purchased from Qualigens and SDFCL Company respectively and used without further purification. CuI particles were prepared by the following experimental sequence - 30 mL of kidney bean seed extract was added drop wise into the copper nitrate solution under magnetic stirring, and then the sodium iodide solution was added drop wise into the above solution. The obtained mixture was stirred at room temperature for 30 min. The resultant dark brown precipitate was filtered, washed thoroughly with 50% ethanol and dried at 50o C.

3 Results and Discussion:

CuI nanoparticles were synthesized using seed extract / fruit juice by the method explained in the experimental section. 30ml of fruit juice/ seed extract for synthesis of CuI was optimized after trying out different t volumes (5 - 40ml). It was found that the yield was maximum for 30ml of extract. It was observed that the texture of recipitate obtained was thick and uniform. The synthesized precipitates of CuI were characterized using X-ray powder diffractogram, SEM and EDAX techniques.

3.1 X-ray powder diffracton

The X-ray powder patterns of the CuI nanoparticles synthesized from kidney bean seed extract is represented in Figure 1. It was observed that the x-ray powder data matched well with that reported in [2] (JCPDScardNo.82–2111). CuI was found to crystallize in cubic phase with a space group of F-43m . No significant impurities were observed in the XRD patterns, indicating high purity of the products.

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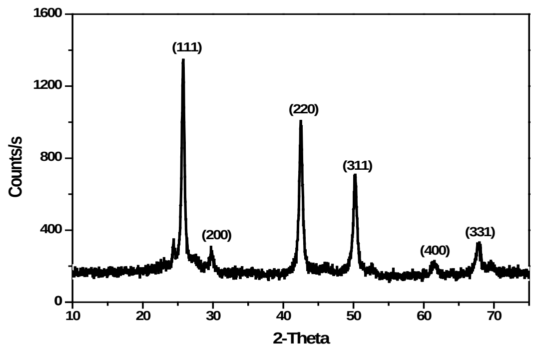


Figure 1: XRD patterns of the nanoparticles synthesized by kidney bean seed extract

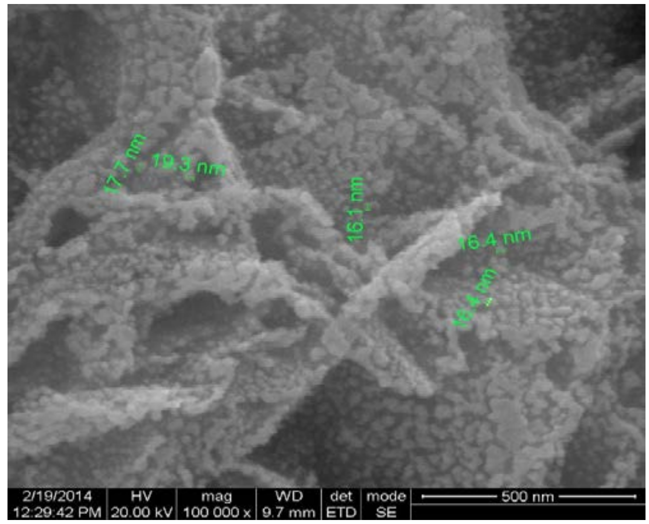
All the reflections correspond to pure CuI particles with face centered cubic symmetry. The high intense peak for fcc materials is generally (1 1 1) reflection, which is observed in the sample. The intensity of peaks reflected the high degree of crystallinity of the synthesized products. However, the diffraction peaks are broad which may be attributed to the small crystallite size. Using Scherer formula the average particle size of CuI particles was found to be 20.2028 nm.

2 Theta	Rel.Int . [%]	Crystal Size nm	d-spacing Å	(hkl)	a Å
25.7626	100.00	23.14	3.45817	(111)	5.98972
29.8248	7.26	16.05	2.99577	(200)	5.99154
42.5415	65.25	38.03	2.12511	(220)	6.01072
50.2898	42.71	27.4	1.81436	(311)	6.01755
52.6942	2.68	11.54	1.73710	(222)	6.01749
61.5514	5.36	10.31	1.50668	(400)	6.02672
67.8381	11.25	14.95	1.38155	(331)	6.02203
		Mean = 20.2028			Mean a = 6.0108

Table 1: Peak position and crystallite size CuI NPs synthesized from kidney bean extract

3.2 SEM and EDAX morphological studies of CuI Nanoparticles

The SEM pictures of C ()uI nanoparticles synthesized using kidney bean seeds. They appear to be spheres made of flakes. The Figure 2 and 3 shows the same. The particles seem to have flower like structures. This fact is complimented by the broader peaks in the X-ray powder pattern of CuI from Kidney bean seed extract The mean particle size of CuI from Kidney bean seed extract was found to be 28.12 nm. EDAX shown in figure 3of synthesized nanoparticles show that they are made of only pure CuI.



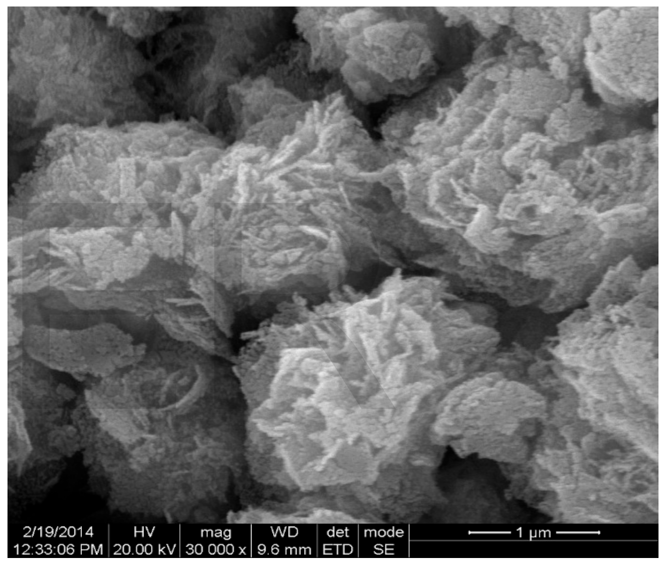


Figure 2: SEM image of the CuI using kidney bean seed extract

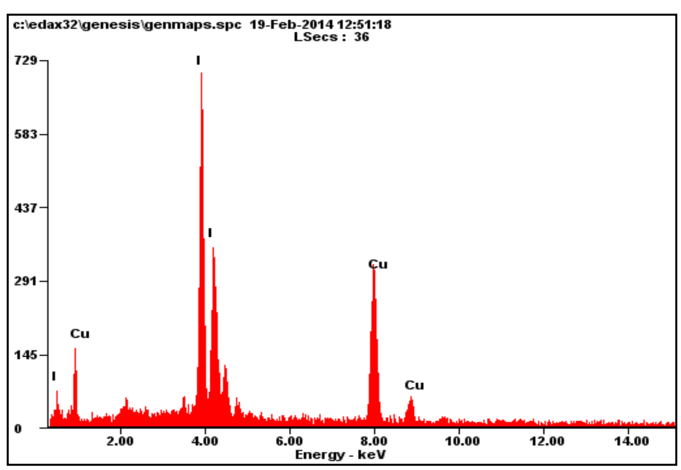


Figure 3. EDAX of the CuI using kidney bean seed extract

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3.3 Antibacterial activity

If a bacterium is susceptible to a particular nanoparticle, a clear area surrounds the substance where bacteria are prevented from growing (called a zone of inhibition).

S.No	Name of bacteria	Variety of bacteria	Diameter zone of inhibition for black kidney bean seed (in mm)
1.	Escherichia coli	Gram (-)	14
2.	Staphylococcus aureus	Gram (+)	10
3.	Klebsiella pneumoniae	Gram (-)	13
4.	Proteus mirabilis	Gram (-)	7
5.	Bacillus cereus	Gram (+)	13

Table 2: Comparison of activities of Copper iodide Nanoparticles on Gram (-) & Gram (+) bacteria

The synthesized CuI NPs was tested for antibacterial sensitivity using the surface inoculation method and broth inoculation method. It was observed that synthesized CuI showed antibacterial activity. (Supplementary data)

4. Conclusion

It was observed from the XRD data that all the reflections corresponded to pure CuI nanoparticles with face centered cubic symmetry with a space group of F43m. The intensity of peaks reflected the high degree of crystallinity of the synthesized products. However, the diffraction peaks were broad which indicated that the crystallite size is very small.

From the SEM data, it was observed that the particle size of CuI synthesized using Kidney bean seeds extract were smaller in size. This fact was complimented by the broader peaks in the X-ray powder pattern of CuI from Kidney bean seed. EDAX analysis indicated that the composition of copper and iodide were in the ratio of 1:1 confirming the formation of Copper (I) iodide.

CuI synthesized is sensitive against bacteria. It is observed that the CuI synthesized from kidney bean seed extract shows a very good sensitivity

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Supplementary Data

Green Synthesis and Characterisation of Copper (I) Iodide nanoparticles using kidney bean seed extract and its anti-bacterial activity

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1. ANTIBACTERIAL ACTIVITY:

The following data shows the antibacterial activity for copper iodide nanoparticles synthesized using kidney bean seed extract (CuI-S) and CuI prepared using pomegranate juice extract (CuI-P).

If a bacterium is susceptible to a particular nanoparticle, a clear area surrounds the substance where bacteria are prevented from growing (called a zone of inhibition). The synthesized CuI NPs was tested for antibacterial sensitivity using the surface inoculation method and broth inoculation method.

S.No	Name of bacteria	Variety of bacteria	Diameter zone of inhibition for black kidney bean seed (in mm)
1.	Escherichia coli	Gram (-)	14
2.	Staphylococcus aureus	Gram (+)	10
3.	Klebsiella pneumoniae	Gram (-)	13
4.	Proteus mirabilis	Gram (-)	7
5.	Bacillus cereus	Gram (+)	13

Table 2: Comparison of activities of Copper iodide Nanoparticles on Gram (-) & Gram (+) bacteria

1.1 Surface inoculation method:

The Figure 4 shows the inhibition of growth of Escherichia coli on the inoculated medium around the CuI NPs. Here, substance 1 is CuI NPs synthesized from pomegranate extract (CuI-P) and substance 2 is the CuI NPs synthesized from kidney bean seed extract (CuI-S).

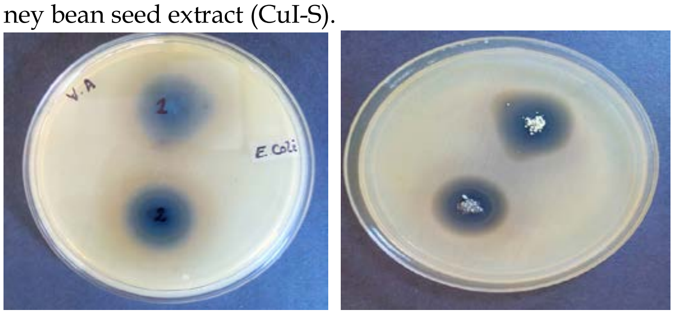
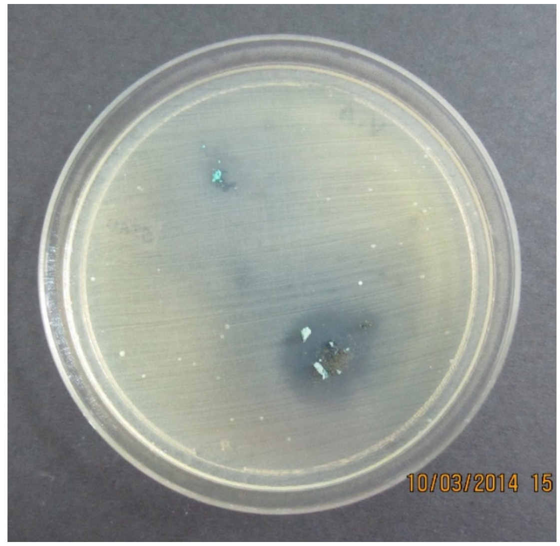


Figure 4: Diameter zone of inhibition against Escherichia coli Substance 1- 25 mm and Substance 2- 14 mm

The figure 5 shows the inhibition of growth of Staphylococcus aureus on the inoculated medium around the CuI NPs.



Fi gure 5: Diameter zone of inhibition against Staphylococcus aureus Substance 1- 3 mm and Substance 2-10 mm

The figure 6 shows the inhibition of growth of Klebsiella pneumoniae on the inoculated medium around the CuI NPs.

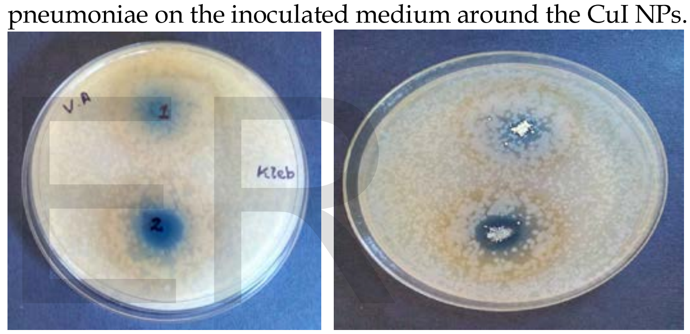


Figure 6: Diameter zone of inhibition against Klebsiella pneumoniae Substance 1- 12 mm and Substance 2- 13 mm

The figure 7 shows the inhibition of growth of Proteus mirabilis on the inoculated medium around the CuI NPs.

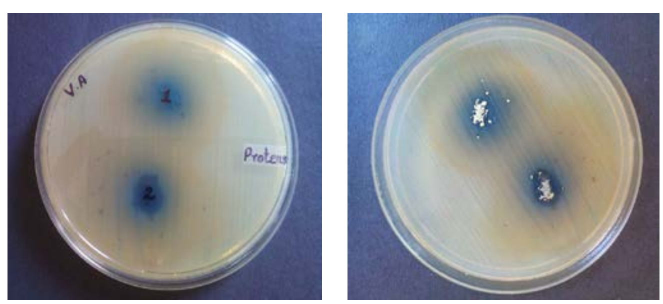


Figure 7: Diameter zone of inhibition against Proteus mirabilis Substance 1- 6 mm and Substance 2- 7 mm

The figure 8 shows the inhibition of growth of Bacillus cereus on the inoculated medium around the CuI NPs.

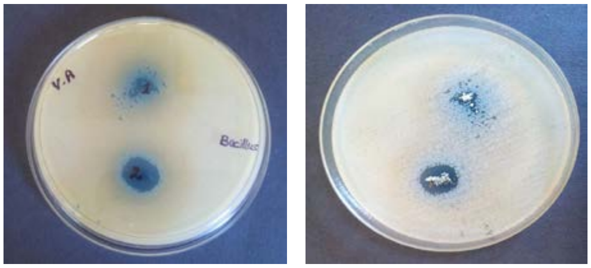


Figure 8: Diameter zone of inhibition against Bacillus cereus Substance 1- 10 mm and Substance 2- 13 mm

1.2 Broth inoculation method

Antibacterial activity of CuI- P

Escherichia coli

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 3.

Time (hrs)	Control (Broth + E.coli)	Test (Broth + E.coli + CuI-P)
0	0	0
18	0.42	0.09
19	0.36	0.18
21	0.32	0.2
23	0.33	0.23

Table 3: Optical density readings of CuI-P on E.coli

Increase in bacterial growth is seen in both control and test, the higher absorbance values in test sample shows the decreased growth of E.coli in the presence of the nanoparticle.

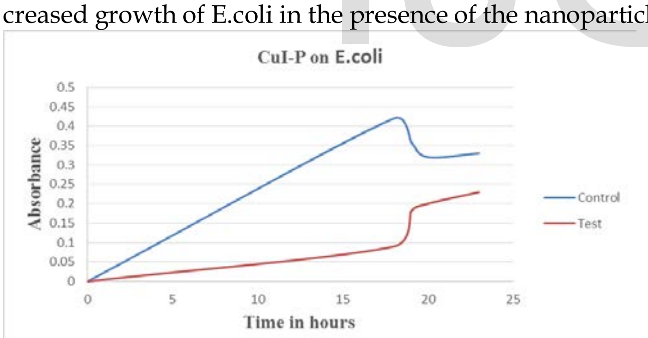


Figure 9: Antibacterial activity of CuI-P on E.coli

Staphylococcus aureus

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 4.

Time (hrs)	Control (Broth + S.aureus)	Test (Broth + S.aureus + CuI-P)
0	0	0
18	0.13	0.05
19	0.15	0.14
21	0.11	0.09
23	0.17	0.14

Table 4: Optical density readings of CuI-P on S.aureus

The absorbance obtained shows an increase in control and there is inhibition seen in the test. The inhibition in the test is due to the minimal suspension of nanoparticles used, Increase in bacterial growth is seen in both control and test, the lower absorbance values in test sample shows the decreased growth of S.aureus in the presence of the nanoparticle.

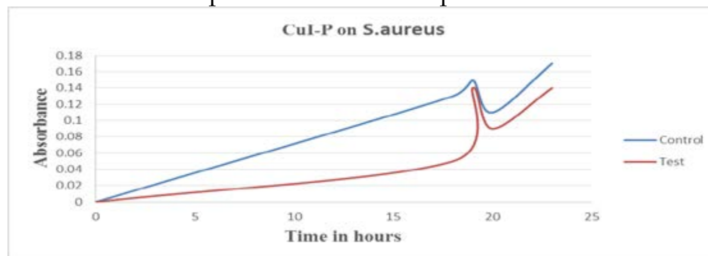


Figure 10: Antibacterial activity of CuI-P on S.aureus

Klebsiella pneumoniae

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 5.

Time (hrs)	Control (Broth + K.pneumoniae)	Test (Broth + K.pneumoniae + CuI-P)
0	0	0
18	0.08	0.04
19	0.14	0.05
21	0.16	0.06
23	0.2	0.05

Table 5: Optical density readings of CuI-P on K.pneumonia

The absorbance obtained shows an increase in control and there is inhibition seen in the test, Increase in bacterial growth is seen in both control and test, the lower absorbance values in test sample shows the decreased growth of K.pneumoniae in the presence of the nanoparticle.

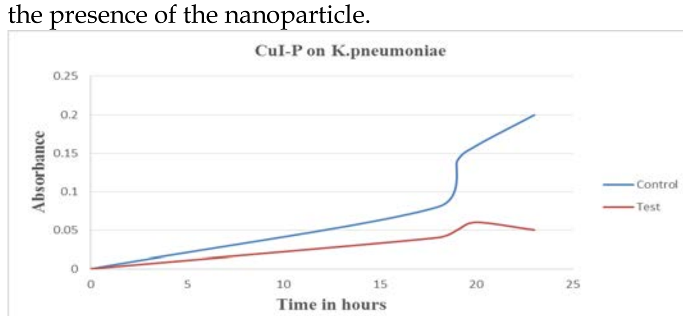


Figure 11: Antibacterial activity of CuI-P on K.pneumonia

Proteus mirabilis

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 6.

An increase in bacterial growth is seen in both samples; the lower absorbance values for the test sample tube indicates the decreased growth of P.mirabilis in the presence of the nanoparticle.

Time (hrs)	Control (Broth + P.mirabilis)	Test (Broth + P.mirabilis + CuI-P)
0	0	0
18	0.14	0.06
19	0.14	0.12
21	0.15	0.14
23	0.16	0.16

Table 6: Optical density readings of CuI-P on P.mirabilis

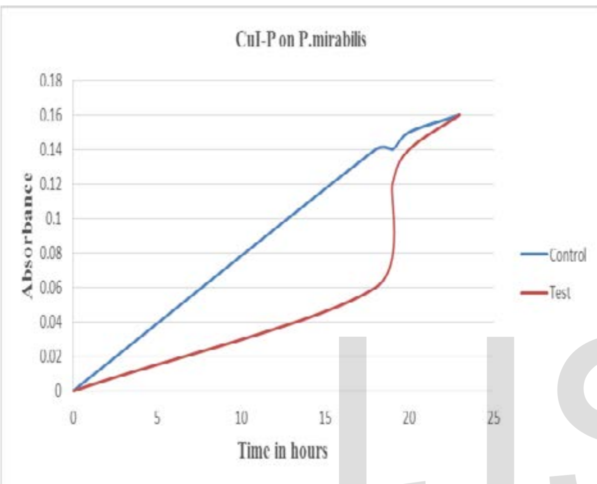


Figure 12: Antibacterial activity of CuI-P on P.mirabilis

Bacillus cereus

Time (hrs)	Control (Broth + B.cereus)	Test (Broth + B.cereus+ CuI-P)
0	0	0
18	0.08	0.04
19	0.11	0.09
21	0.19	0.12
23	0.06	0.05

Table 7: Optical density readings of CuI-P on B.cereus

The absorbance reading of the inoculated bacterial suspension of both control and test is shown below in the Table 7.
Increase in bacterial growth is seen in both control and test, the lower absorbance values in test sample shows the decreased growth of B.cereus in the presence of the nanoparticle shows lesser inhibitory effect of the bacteria.

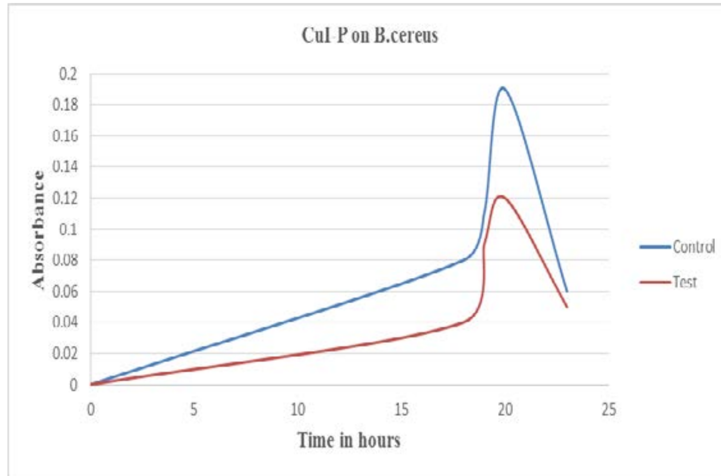


Figure 13: Antibacterial activity of CuI-P on B.cereus

Antibacterial activity of CuI-S.

Escherichia coli

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 8.

Time (hrs)	Control (Broth + E.coli)	Test (Broth + E.coli + CuI-S)
0	0	0
18	0.42	0.1
19	0.26	0.2
21	0.26	0.23
23	0.31	0.3

Table 8: Optical density readings of CuI-S on E.coli

An increase in bacterial growth is seen in both samples; the lower absorbance values for the test sample tube indicate much the decreased growth of E.coli in the presence of the nanoparticle.

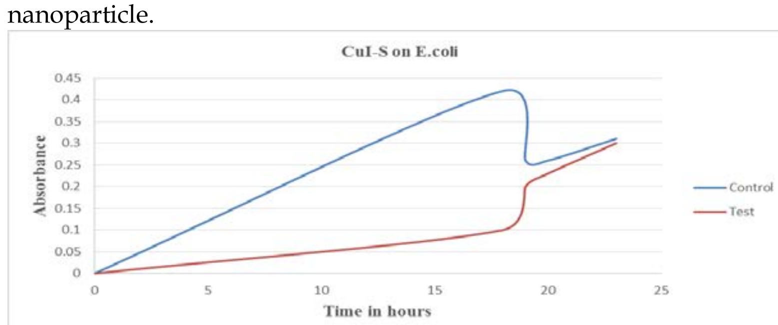


Figure 14: Antibacterial activity of CuI-S on E.coli

Staphylococcus aureus

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The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 9.

Time (hrs)	Control (Broth + S.aureus)	Test (Broth + S.aureus + CuI-S)
0	0	0
18	0.09	0.06
19	0.11	0.07
21	0.16	0.09
23	0.17	0.17

Table 9: Optical density readings of CuI-S on S.aureus

An increase in bacterial growth is seen in both samples; the lower absorbance values for the test sample tube indicate much the decreased growth of S.aureus in the presence of the nanoparticle.

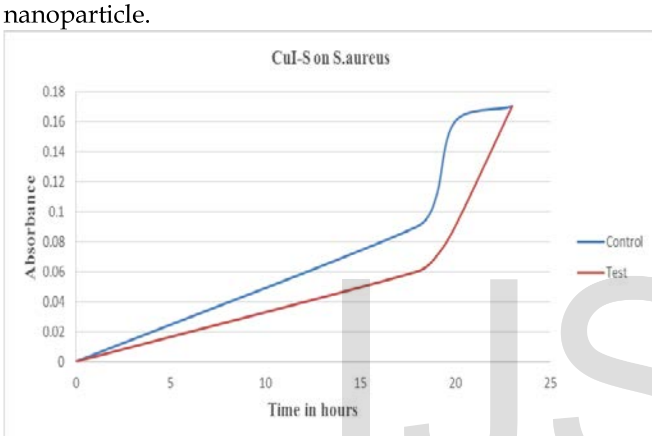

Figure 15: Antibacterial activity of CuI-S on S.aureus

Klebsiella pneumoniae

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 10.

Time (hrs)	Control (Broth + K.pneumoniae)	Test (Broth + K.pneumoniae + CuI-S)
0	0	0
18	0.62	0.11
19	0.1	0.09
21	0.08	0.01
23	0.05	0.01

Table 10: Optical density readings of CuI-S on K.pneumoniae

An increase in bacterial growth is seen in both samples; the lower absorbance values for the test sample tube indicate the decreased growth of K.pneumoniae in the presence of the nanoparticle.

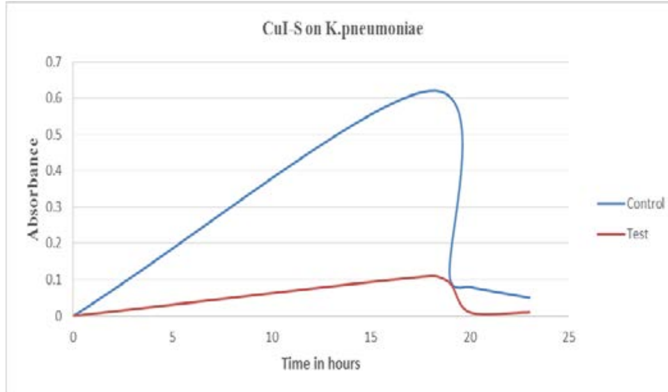

Figure 16: Antibacterial activity of CuI-S on K.pneumoniae

Proteus mirabilis

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 11.

An increase in bacterial growth is seen in both samples, the lower absorbance values for the test sample tube indicate the decreased growth of P.mirabilis in the presence of the nanoparticle.

Time (hrs)	Control (Broth + P.mirabilis)	Test (Broth + P.mirabilis + CuI-S)
0	0	0
18	0.13	0.06
19	0.26	0.14
21	0.15	0.14
23	0.15	0.14

Table 11: Optical density readings of CuI-S on P.mirabilis

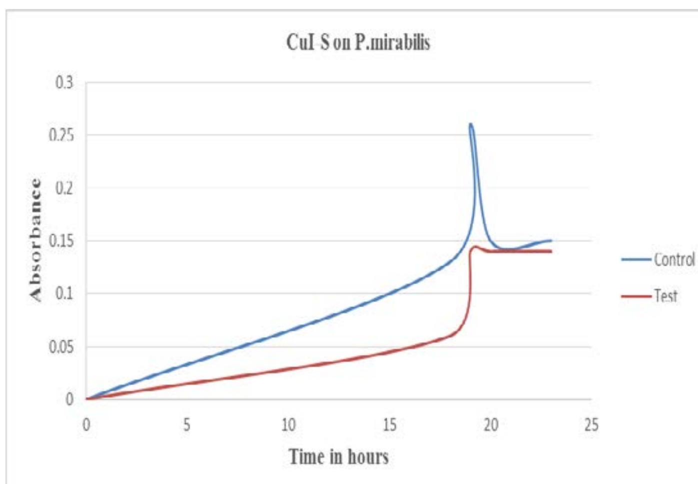

Figure 17: Antibacterial activity of CuI-S on P.mirabilis

Bacillus cereus

The absorbance reading of the inoculated bacterial suspension of both control and the test is shown below in the Table 12.

Time (hrs)	Control (Broth + B.cereus)	Test (Broth + B.cereus + CuI-S)
0	0	0
18	0.08	0.07
19	0.1	0.07
21	0.14	0.02
23	0.16	0.01

Table 12: Optical density readings of CuI-S on B.cereus

An increase in bacterial growth is seen in both samples; the lower absorbance values for the test sample tube indicate much the decreased growth of B.cereus in the presence of the nanoparticle.

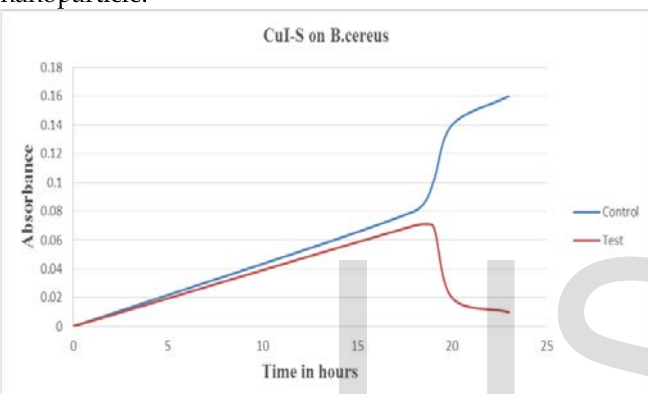

Figure 18: Antibacterial activity of CuI-S on B.cereus

The synthesized CuI NPs was tested for antibacterial sensitivity using the surface inoculation method and broth inoculation method. It was observed that synthesized CuI showed anti-bacterial activity.