Reviewer: 1

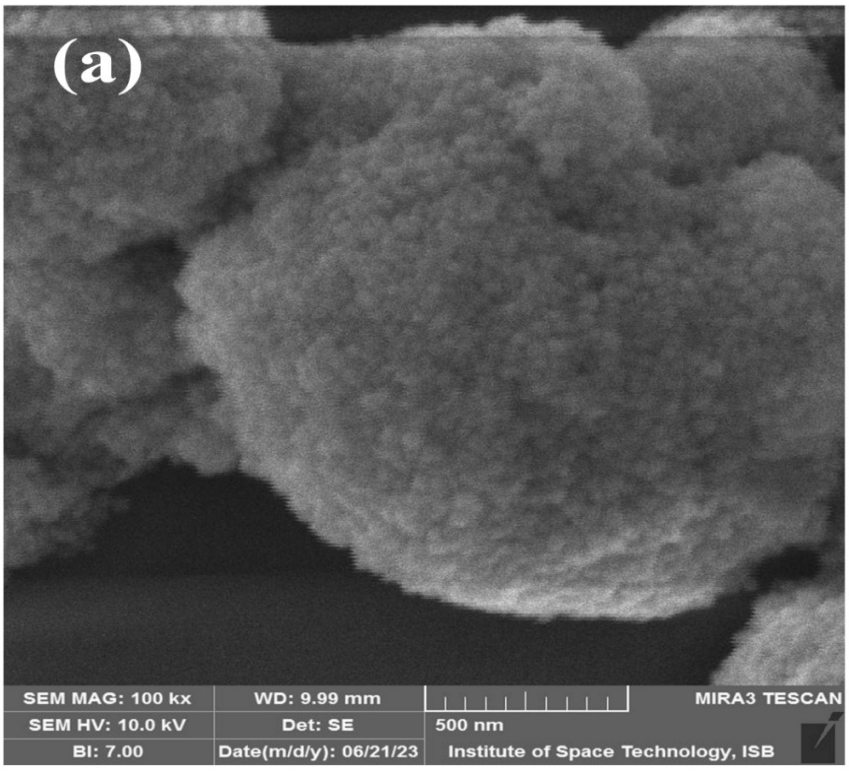
Recommendation: Major revisions

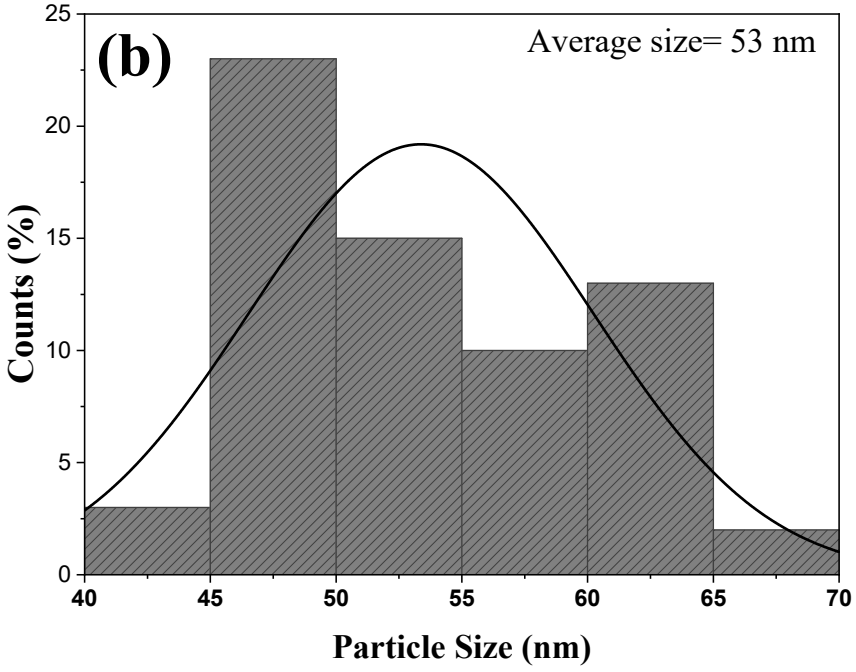
Comments

3. Lack of characterization data for synthetic nano-silver.

Response: Thanks to the Reviewer for raising an important question. SEM analysis is used to characterize the synthetic silver nanoparticles. The results of SEM analysis are as follows:

"SEM analysis is used to study the surface morphology along with size of silver nanoparticles and shown as **Fig. 1(a)** which indicates that Ag NPs are spherical in shape leading to the mono-dispersity with the average size of 53 nm **Fig. 1(b)** Aggregation is due to the refined form of Ag NPs of intermediate size from 45-65 nm. The surface is smooth with well-defined crystalline structure. Elemental analysis through EDX as **Fig. 1(c)** shows that weight percentage of silver is 85.38 % without any contaminants, making it suitable for the enhancement of SERS signal."


(a)
SEM MAG: 100 kx
WD: 9.99 mm
MIRA3 TESCAN
SEM HV: 10.0 kV
Det: SE
500 nm
BI: 7.00
Date(m/d/y): 06/21/23
Institute of Space Technology, ISB


(b)
Average size= 53 nm
Counts (%)
Particle Size (nm)
0
5
10
15
20
25
40
45
50
55
60
65
70

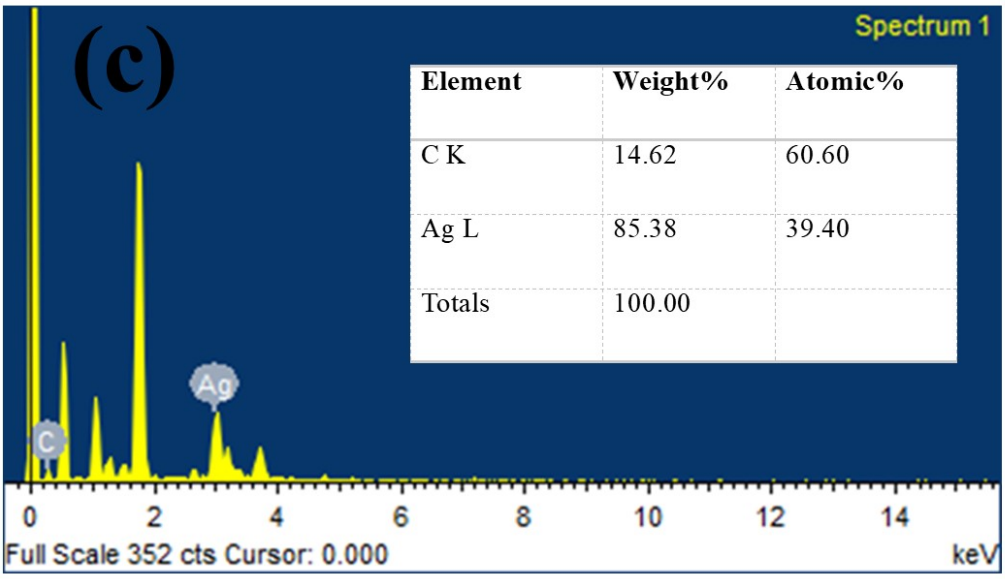


Fig. 1: SEM image of Ag NPs **(a)** Histogram of Ag NPs size **(b)** and elemental analysis of Ag NPs through EDX **(c)**.

Reviewer: 2

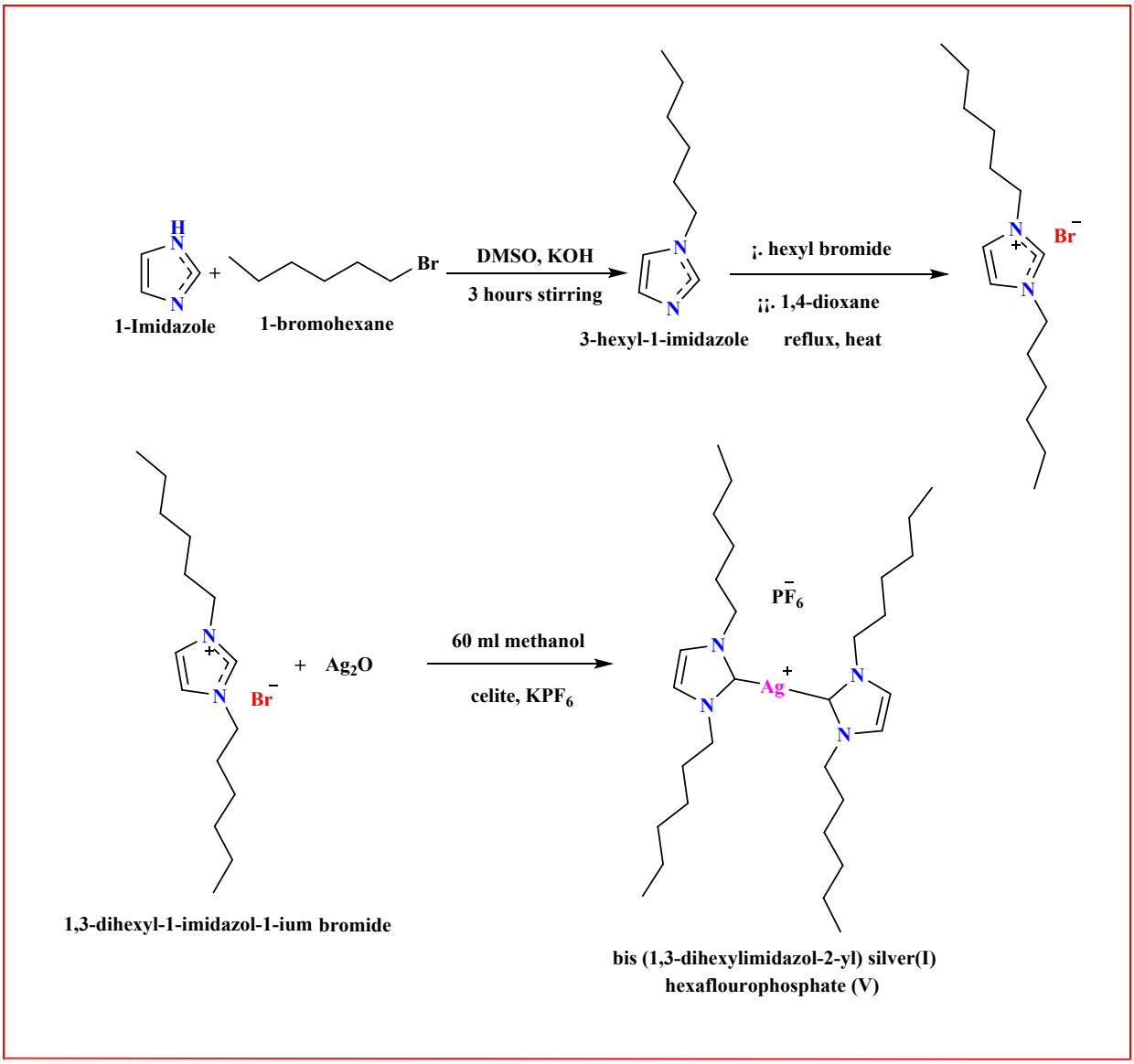
Recommendation: Minor revisions

Comments

2. Section 2.2 takes a large amount of the manuscript but does not really contribute to the methods section. I recommend to remove this discussion or shift it to a supporting information.

2.2. Discussion

The complex is synthesized by using our previously reported method [1,2] with minor modifications. According to this procedure, one equivalent of ligand that has already been synthesized and one equivalent of metal oxide is reacted (**Scheme 1**) [2].



Scheme 1. Synthesis of bis (1,3-dihexylimidazole-2-yl) silver (I) hexafluorophosphate (V).

Since two silver atoms/ions (Ag_2O) are present in a silver oxide molecule, two ligand molecules form a bond with one metal ion. To prepare two units of azolium salt for reaction, the other silver ion and oxygen participate to remove hydrogen from them, making it carbene ready for the reaction. In the flask containing extra AgBr, unreacted Ag_2O, and water, the products appear as a blackish material. To remove undesirable materials from the reaction medium, utilize Celite 545 as a filter and to get the necessary complex, the light yellow to colorless filtrate is evaporated as a thick fluid which can be solidified by changing its counter anion from Br to PF^-_6 metathesis reaction [3].

For the evidence of a successful synthesis of Ag-NHC complexes, FT-NMR characteristic of the complex was analyzed in d_6-DMSO from 0-12 δ ppm for ^{1}H-NMR and 0-210 δ ppm for ^{13}C-NMR studies. We noticed intriguing ^{1}H-NMR characteristics in **Fig. 1**.

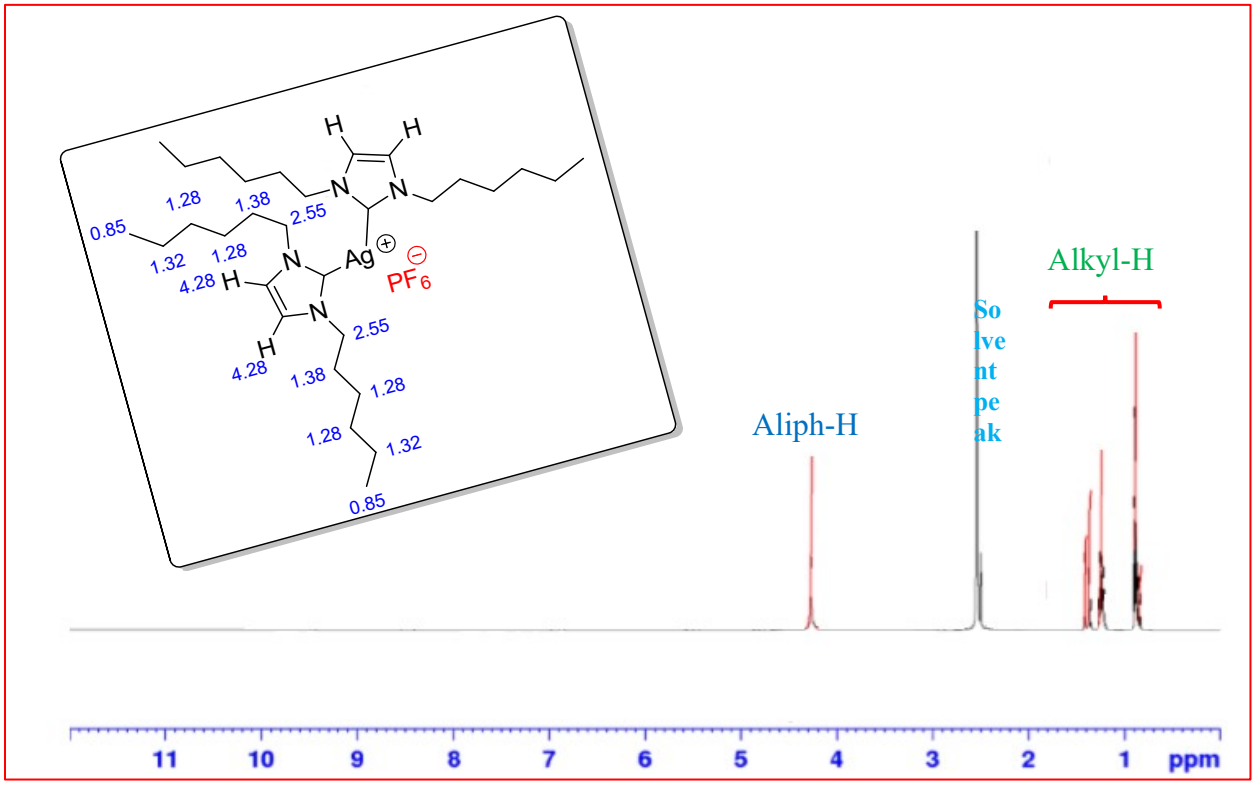


Fig. 1. ^{1}H NMR of bis (1,3-dihexylimidazole-2-yl) silver (I) hexafluorophosphate (V).

In the ^{1}H-NMR spectrum, the Alkyl proton appeared at δ 0.85, 1.28, 1.32, and 1.38 ppm, and the CH_2-N proton appeared at δ 2.55 ppm. The chemical shift of the Aliph-H proton appeared at δ 4.28 ppm. For further confirmation of the structural feature, ^{13}C NMR data has been shown in **Fig. 2.**

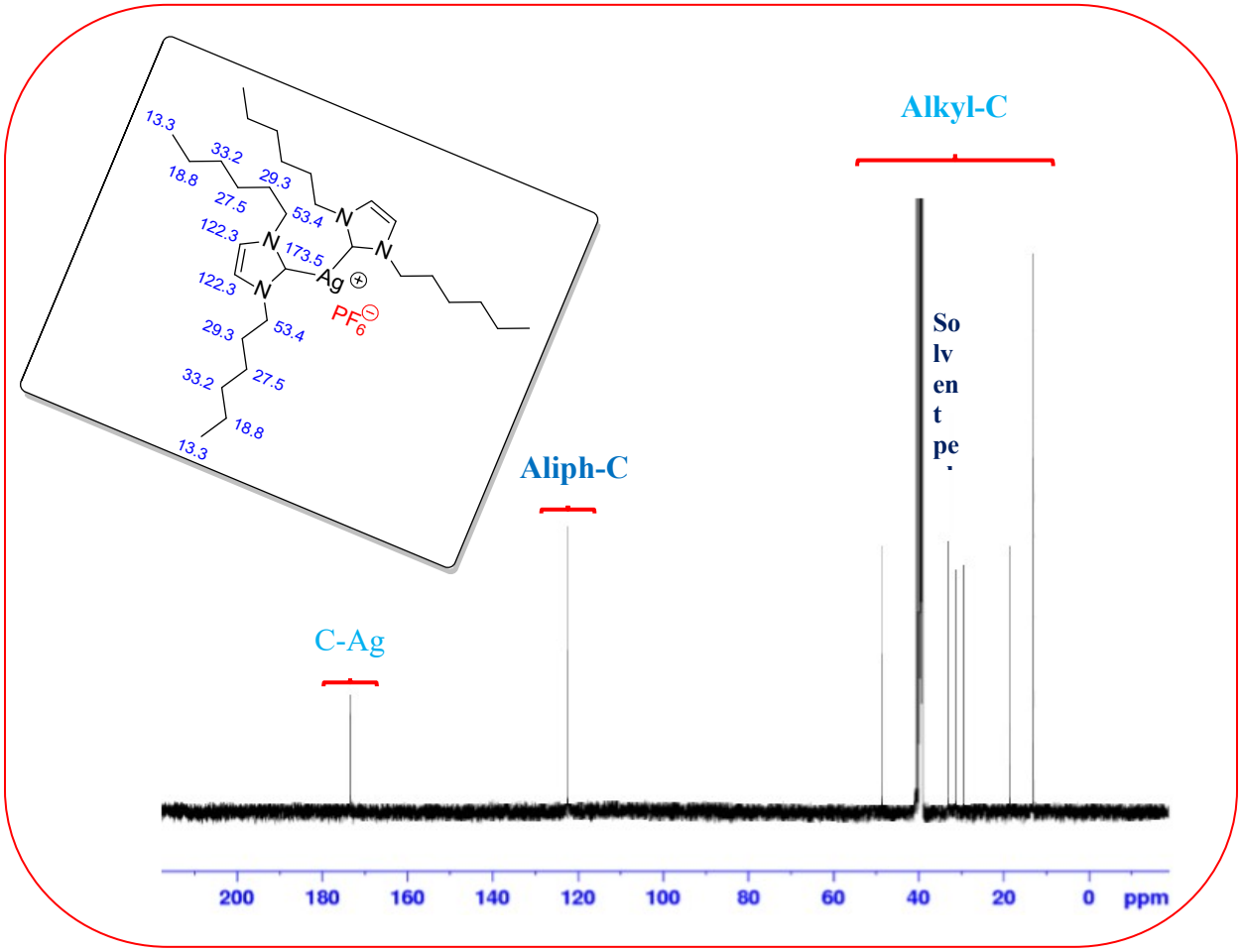


Fig. 2: ^{13}C NMR of bis (1,3-dihexylimidazole-2-yl) silver (I) hexafluorophosphate (V).

According to reported data [1] for comparable azolium salts, the chemical shift value of Alkyl carbon appears at δ 13.3, 18.8, 27.5, 33.2, 48.1 and 53.4 ppm. The chemical shift of Aliphatic carbon appears at δ 122.3 ppm. Upon the silver complexation, imidazole-based Ag complex, the chemical shift appears at *ca* δ 173.5 ppm comparable with the previous reports [2, 4, 5].

References

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