Project Presentation

Wet Impregnation

Wet Impregnation	Incipient Wetness Impregnation (IWI)
More than pore volume (excess liquid)	Exactly equal to total pore volume
Metal precursor soaks and diffuses	Precise control over metal loading
Easier to handle but less precise metal loading	More controlled, but requires good knowledge of support porosity

The amount of water retained within the porous structure of the beads was quantified to determine pore-filling capacity :

figure: beads impregnated in 50 ul ii) beads immersed in 200ul

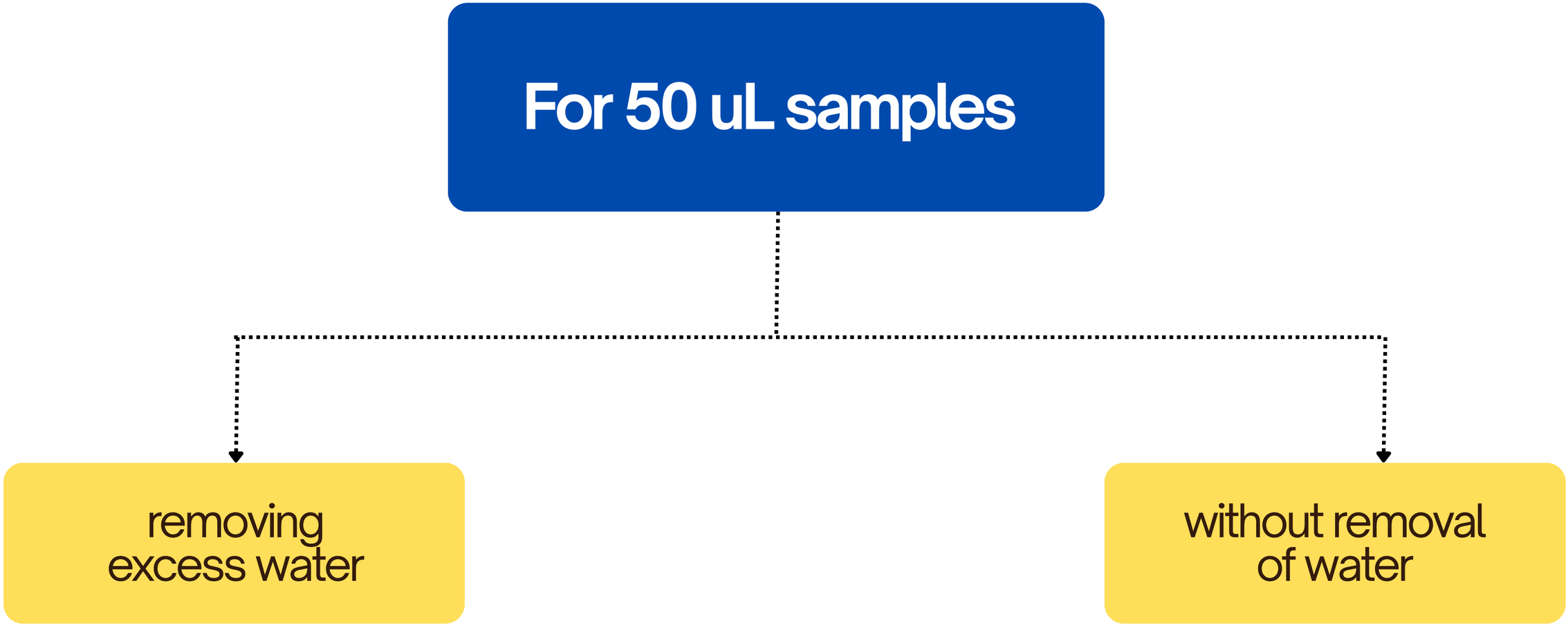
Two conditions were tested using 0.5 g of beads: one with 200 µL of water and another with 50 µL. The volume of water retained within the beads' pores was estimated by comparing the remaining free water after contact.

For 200uL samples : (IWI)

Three samples, each containing 0.5 g of beads, were tested using 200 µL of water. The beads were immersed in water for 30 minutes. After the incubation period, the remaining water was removed using a pipette and weighed. The difference between the initial and final water weight was used to determine the amount of water taken into the pores of the beads.

liq amount :200ul					
viel wt(g)	beads wt(g)	total intial	final wt(g)	difference(g)	
3.52	0.5024	4.0224	4.05	0.0276	
3.48	0.503	3.983	4.003	0.02	
3.6279	0.5006	4.1285	4.1557	0.0272	
			Std deviat	0.004277	0.0043
			average	0.024933	0.025
for 1 g of beads = 0.050					
density =	1000				
g to ul	25	ul			

Mean: 0.025
standard deviation: 0.0043


For 50 uL samples
removing excess water
without removal of water

For 50uL samples : (Wet Impregnation)

Without removal of water

viel wt(g)	beads wt(g)	total intial	final wt(g)	difference(g)
3.873	0.5052	4.3782	4.47	0.0918
3.517	0.5028	4.0198	4.1132	0.0934
3.6014	0.5022	4.1036	4.2	0.0964
mean	0.093867			
std deviation		0.002335		

Mean: 0.0939
standard deviation: 0.0023

For 50uL samples :

Removal of water

after water removal		difference
4.4		0.0218
4.05		0.0302
4.1302		0.0266
mean	0.0262	
std deviation		0.004214

Mean: 0.0262
standard deviation: 0.0042

Samples

- Surface cleaned with HNO_3 and HNO_3 Used as Substitute for $AgNO_3$
- Surface cleaned with HNO_3
- No Pre-cleaning of Surface Prior to Impregnation

Sample 1

Surface cleaned with HNO_3 and HNO_3 Used as Substitute for $AgNO_3$

WHY???

1) Removal of contaminants & surface impurities

- α-Alumina is chemically inert, but surface layers (oxides, adsorbed ions, organic residues) can impede catalyst loading or ion exchange.
- Nitric acid leaches away these layers, exposing fresh, reactive alumina surfaces.

2) This acidic pre-treatment modifies the surface, enhancing adhesion and dispersion of metal or halide precursors (e.g., AgBr, AgI).

3) Demonstrated enhancement in adsorption performance

Facile method to synthesize efficient adsorbent from alumina by nitric acid activation: Batch scale defluoridation, kinetics, isotherm studies and implementation on industrial wastewater treatment
Usha Kumari[1], Sushanta K Behera[2], Hammad Siddiqi[2], B C Meikap[3]
https://pubmed.ncbi.nlm.nih.gov/31376661/

Coating on Alpha-Alumina Beads – Procedure

- 1 g of alpha-alumina beads was taken.
- Cleaned using 50 µL of HNO_3.
- Beads were impregnated for 30 minutes.
- Washed using a sonicator.
- 50 µL of KBr was added.
- Instead of $AgNO_3$, HNO_3 was used again.
- Beads were exposed under halogen lamp
- washed in DI water

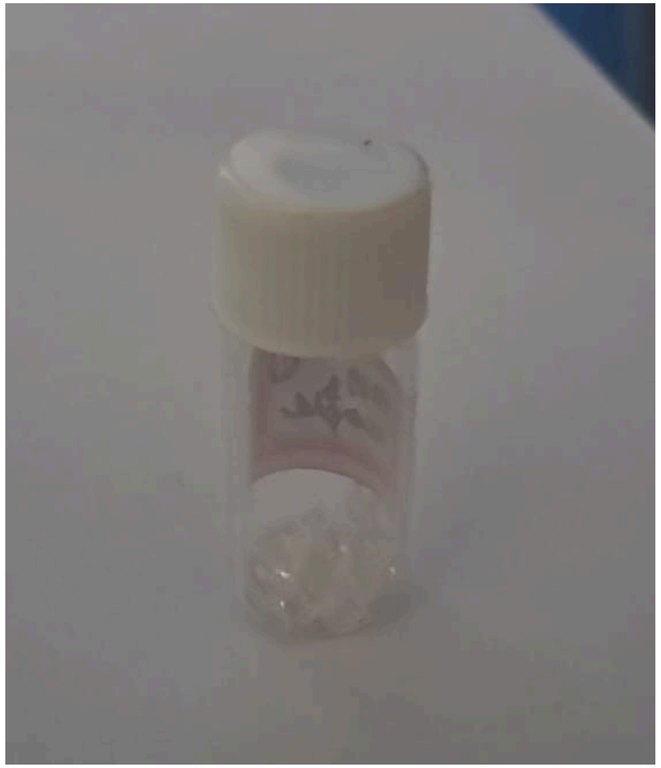


Fig: after impregmenting KBR/KI (50ul)

Fig: after impregmenting HNO3

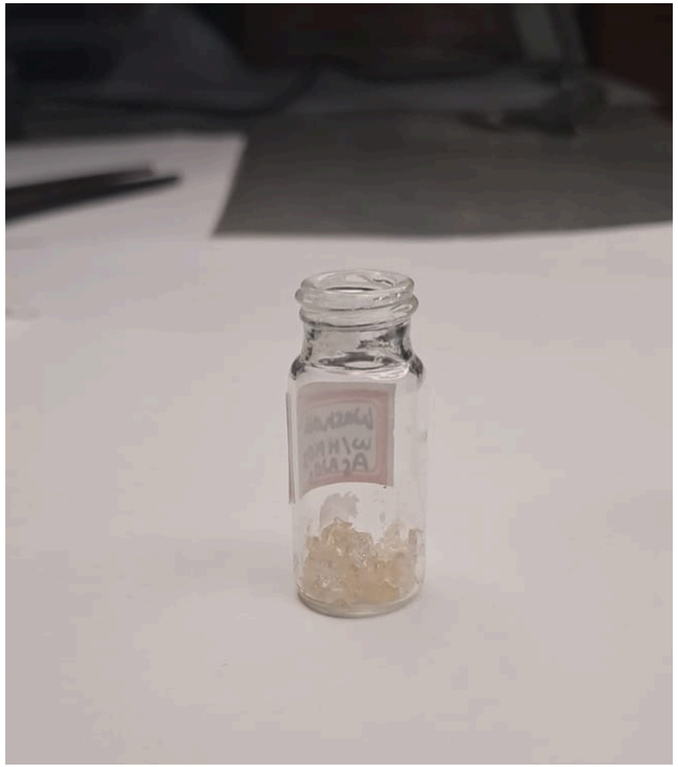


Fig: post exposure and development

Sodium Thiosulphate and Potassium Ferricyanide to Test the Catalyst

- Sodium thiosulphate is a reducing agent, and potassium ferricyanide is an oxidizing agent.
- Normally, their reaction is very slow.
- If our catalyst is active, it speeds up this redox reaction.
- During the reaction, ferricyanide gets reduced to ferrocyanide.
- A clear color change shows that the catalyst is working.

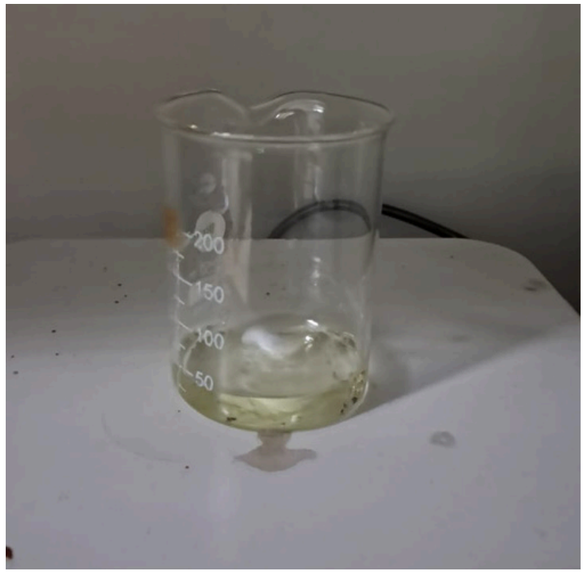
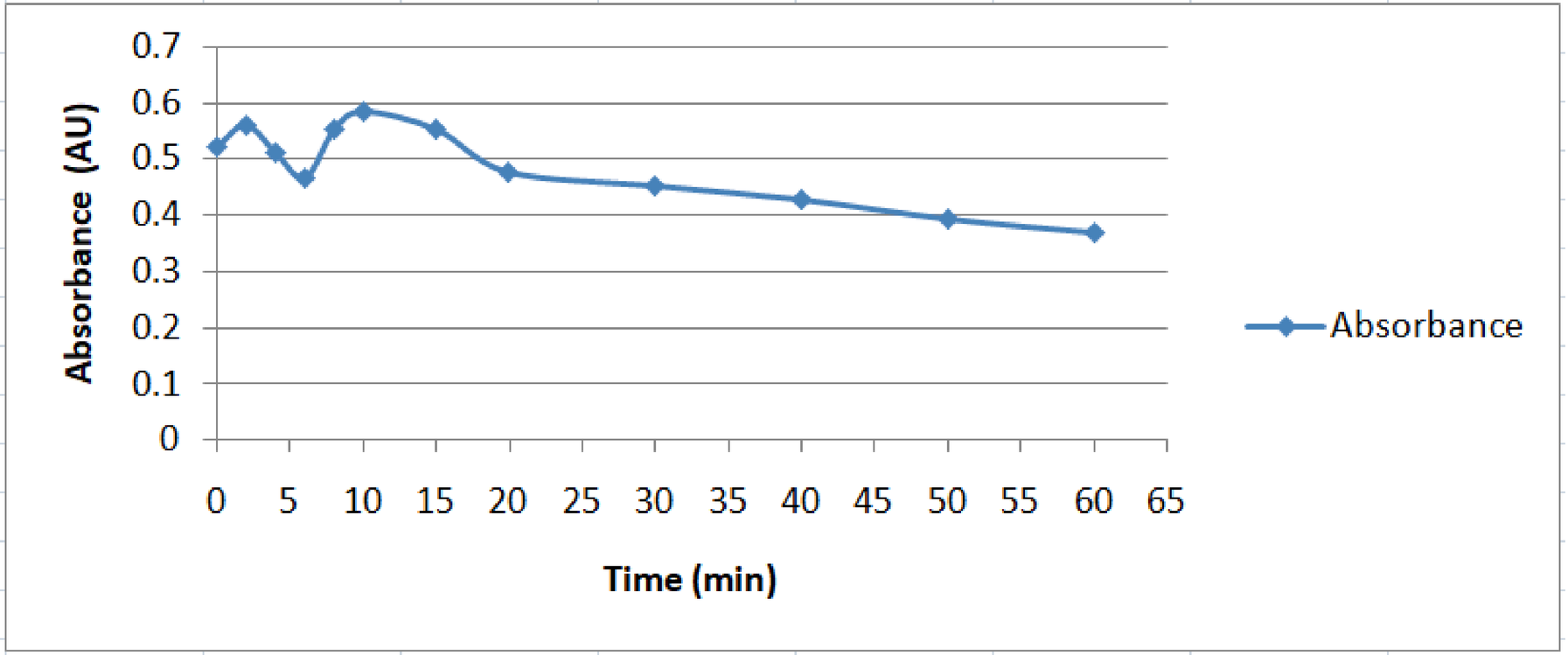


Fig: Sample on top of magnetic stirrier at 350rpm, 40C

UV-Vis Spectrophotometer Result:

	Absorbance
0	0.523
2	0.561
4	0.51
6	0.466
8	0.554
10	0.584
15	0.553
20	0.478
30	0.452
40	0.427
50	0.393
60	0.37



Sample 2

Surface cleaned with HNO_3

Silver Coating on Alpha-Alumina Beads – Procedure

- 1 g of alpha-alumina beads was taken.
- Cleaned using 50 µL of HNO_3.
- Beads were impregnated for 30 minutes.
- Washed using a sonicator.
- 50 µL of KBr was added.
- $AgNO_3$,was used
- Beads were exposed under halogen lamp 10 mins
- Developed and washed in DI

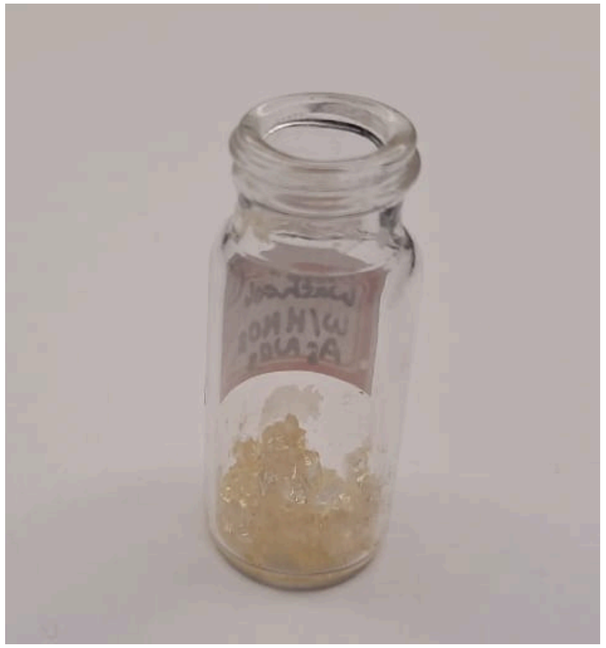


Fig: pretreatment with HNO3

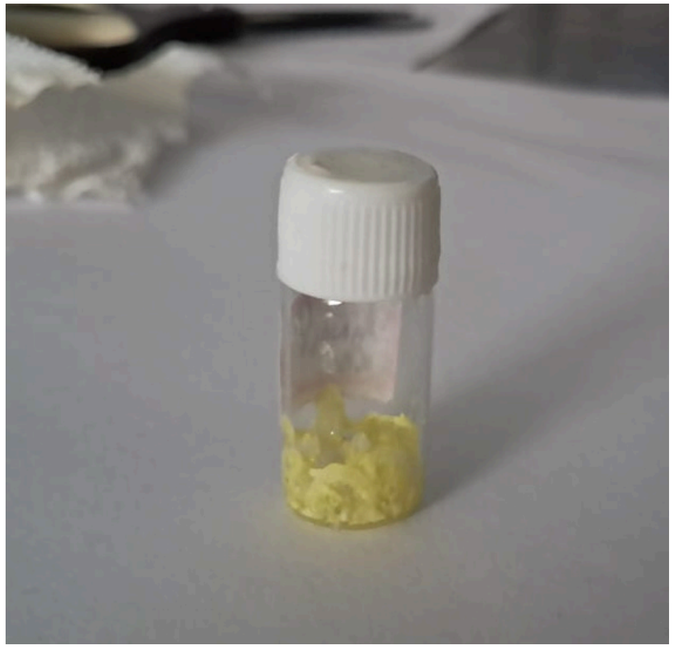


Fig: impregmenting in AgNo3

Fig: post exposure of halogen lamp

Fig: sample after impregmenting in developer solution

Fig:
Sample was subsequently immersed in deionized (DI) water for post-treatment evaluation

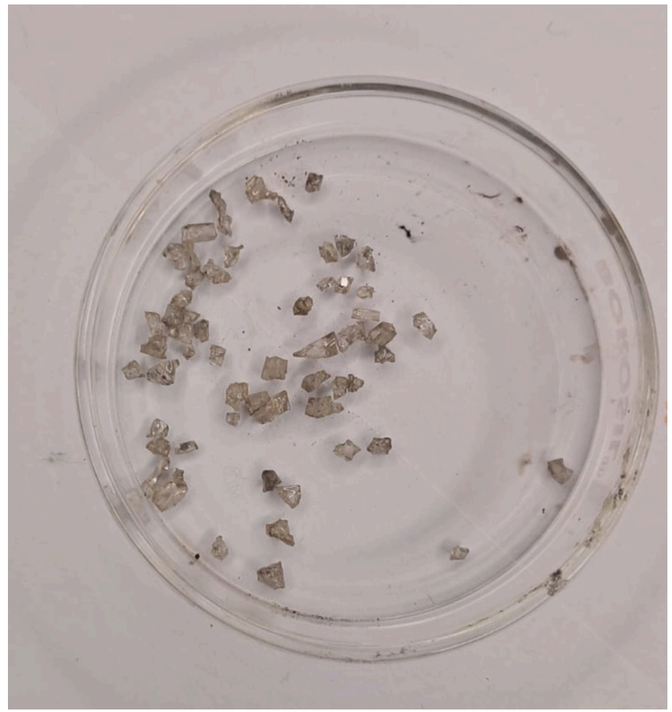
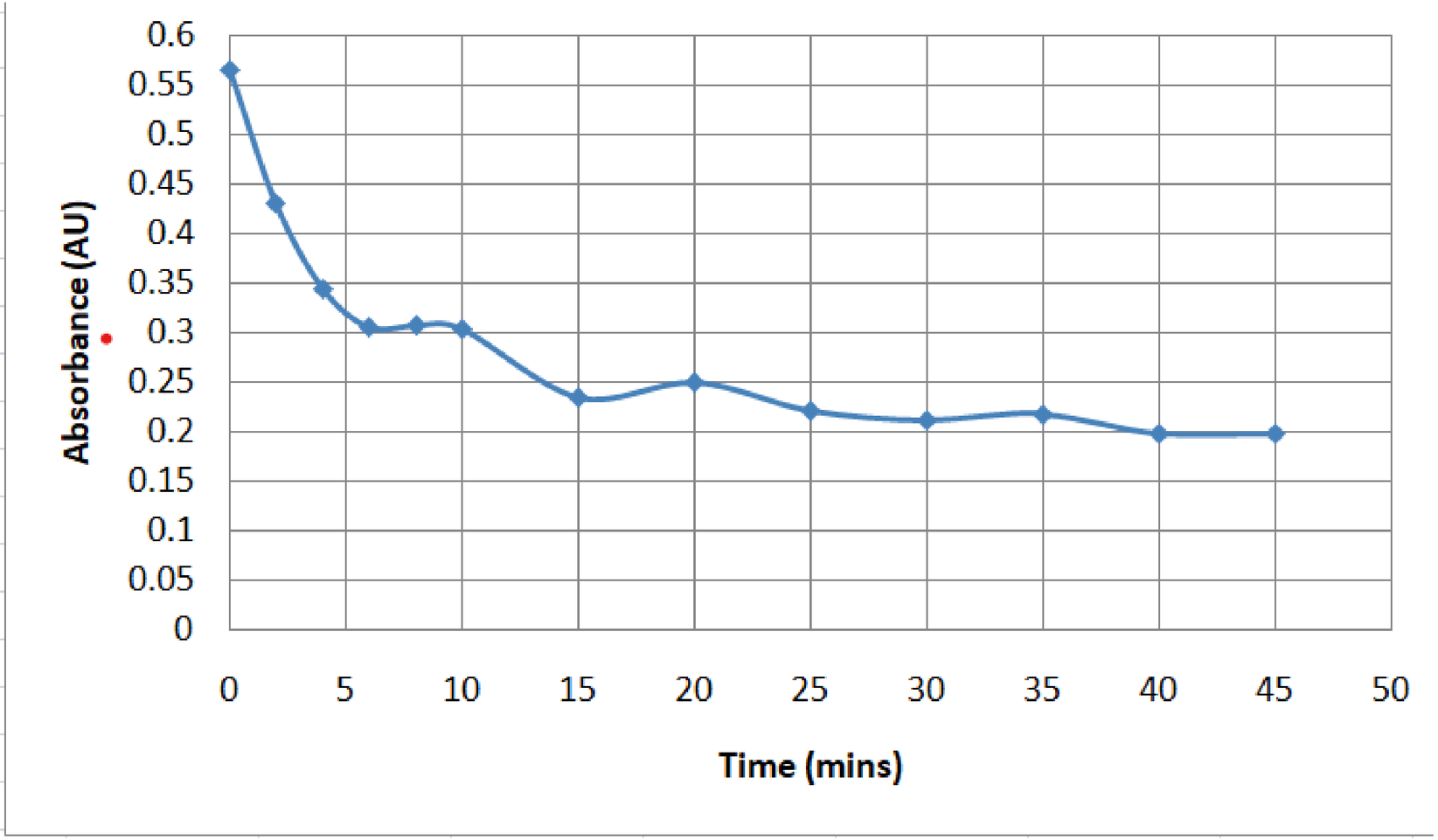


Fig: sample dried at 90 C

UV-Vis Spectrophotometer Result:

	ABSORBANCE (AU)
0	0.564
2	0.431
4	0.344
6	0.306
8	0.308
10	0.304
15	0.235
20	0.249
25	0.222
30	0.212
35	0.218
40	0.199
45	0.199



Sample 3

No Pre-cleaning of Surface Prior to Impregnation

Silver Coating on Alpha-Alumina Beads – Procedure

- 1 g of alpha-alumina beads was taken.
- Beads were impregnated for 30 minutes in KBr/KI(95:5) then dried
- 50 µL of AgNo3 was added.
- Beads were exposed under halogen lamp 10 mins
- Developed and washed in DI

Fig: after impregmenting KBr/KI

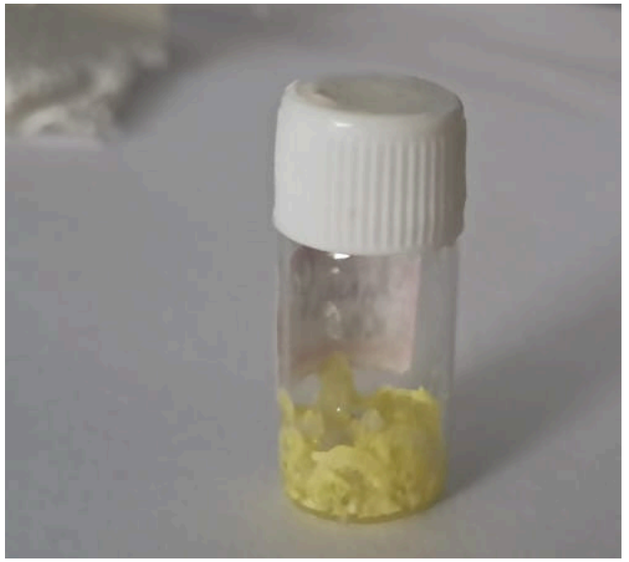
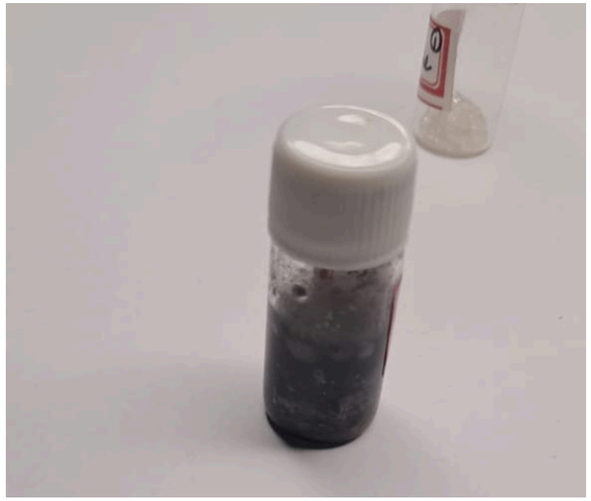


Fig: after impregmenting AgNO3



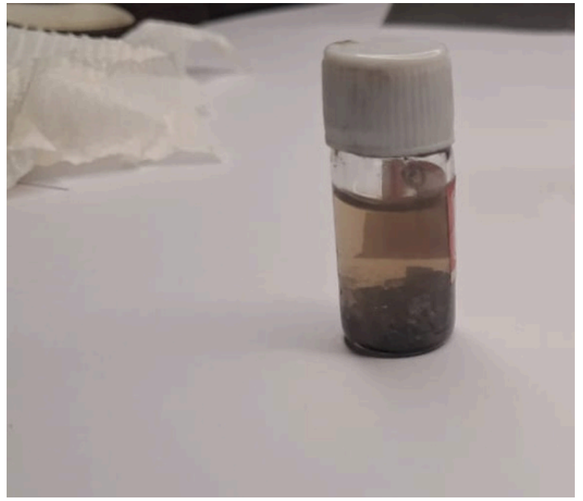


Fig :*Sample was subsequently immersed in deionized (DI) water for post-treatment evaluation*

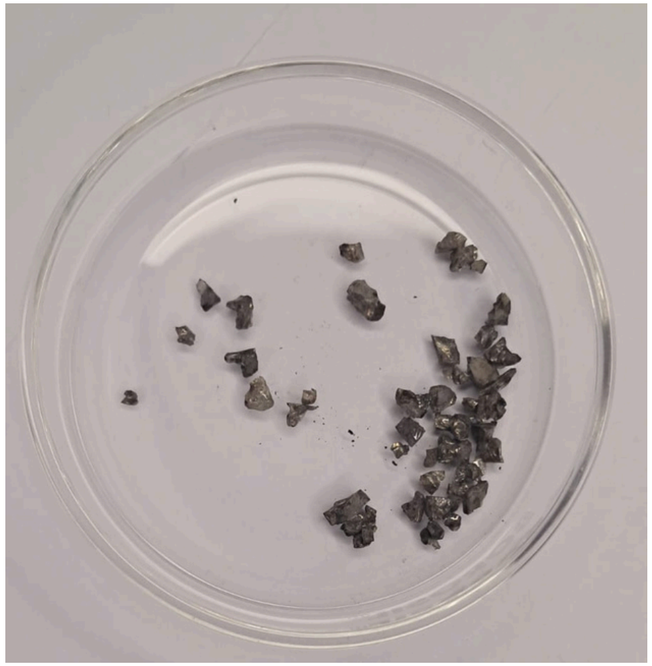


Fig: Sample dried at 90 C

Filling Aluminum Pores with Silver

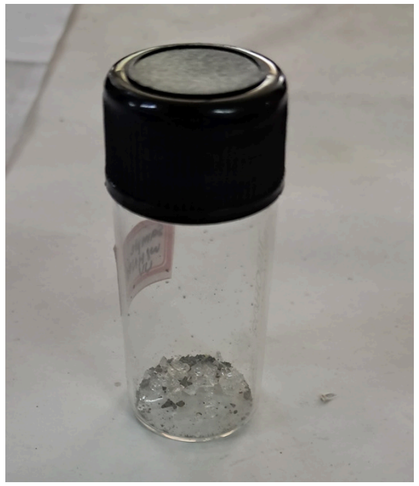
Sample	Pretreatment	KBr:KI Soaking	$AgNO_3$ Soaking	Halogen Lamp	Developer	Ag came out in Developer	Ag came out in DI Water	Remarks
1	None	20 mins (1 mL)	20 mins (1 mL)	10 mins	15 mins	Minimal	Minimal	Double coating used, beads were turned grey with coating
2	KOH (20 mins)	20 mins (1 mL)	20 mins (1 mL)	10 mins	15 mins	Minimal	Minimal	Double coating used, beads were turned grey
3	None	Overnight (2–3 mL)	2 hrs + 20 mins (2–3 mL)	10 mins	15 mins	Almost all Ag came out	Almost all Ag came out	Over-soaking weakened binding
4	HNO3 (30mins)	30mins (0.5ml)	30mins (0.5ml)	10mins	15mins	Ag particles were seen in the solution	Ag particles were seen in the DI water	Slight change in colour of the beads
5	HNO3 (30mins) 1:1 ratio	30mins (0.5ml)	30mins (0.5ml)	**UV LAMP**	15mins	Ag particles were seen in the solution	Ag particles were seen in the DI water	

sample 1

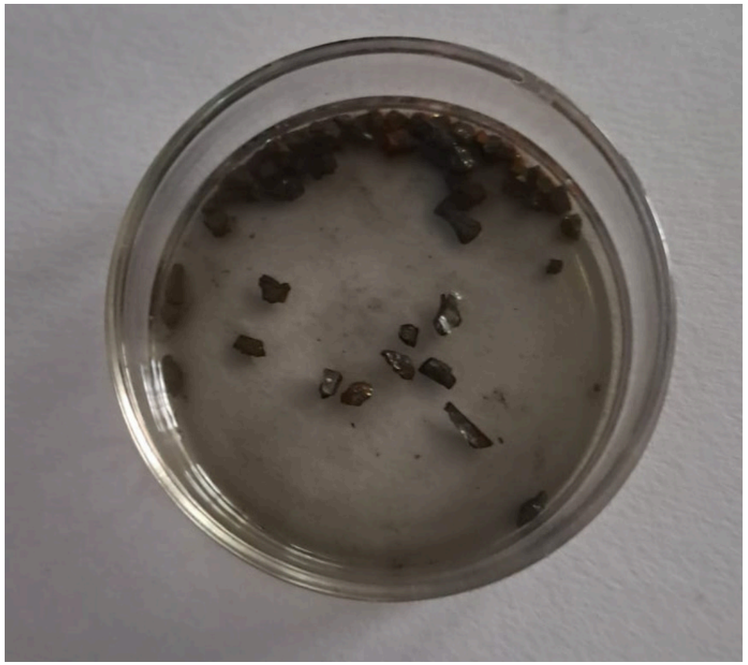
sample 2

sample 3

sample 4

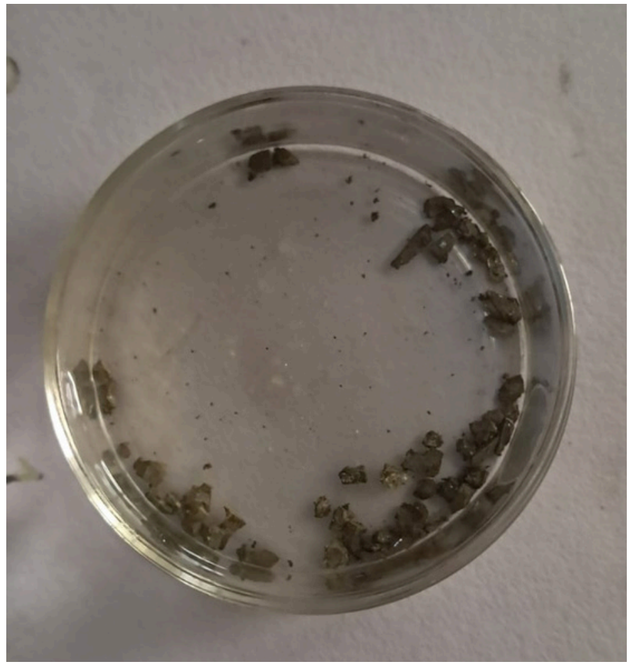


sample 5



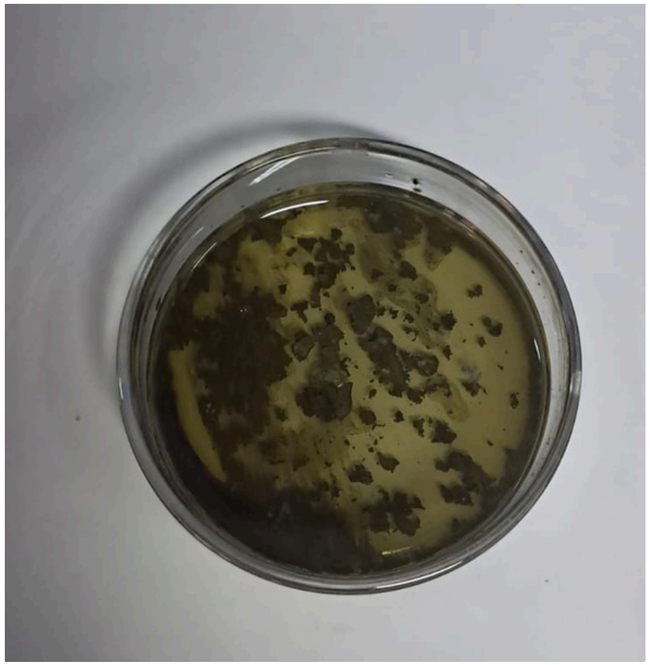
sample 1

Sample 1 was subsequently immersed in deionized (DI) water for post-treatment evaluation



sample 2

Sample 2 was subsequently immersed in deionized (DI) water for post-treatment evaluation



sample 3

Sample 3 was subsequently immersed in developer solution as a result significant amount of silver were getting dispersed in water

UV-Vis Spectrophotometer Result: KOH sample