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Polymorphism, isostructurality and physicochemical properties of glibenclamide salts

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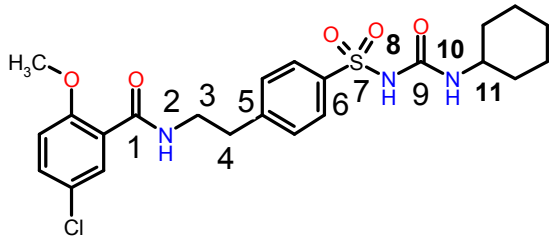
Electronic Supplementary Information†

Table S1 Torsion angles in GBA molecule in salt crystal structures.



τ_1 C1-N2-C3-C4, τ_2 N2-C3-C4-C5, τ_3 C6-S7-N8-C9, τ_4 S7-N8-C9-N10, τ_5 N8-C9-N10-C11

	τ_1	τ_2	τ_3	τ_4	τ_5
GBA	–84.26	178.92	70.10	178.14	178.63
GBA-Na form I	174.84	–47.82	74.19	179.22	172.36
GBA-Na form II	177.75	–47.75	68.35	–172.25	173.28
GBA-Na form III	106.44	57.68	–69.60	–172.57	177.64
GBA-K form I	179.99	47.98	–68.50	171.93	–172.85
GBA-NH_4	–108.62	–40.56	–68.29	172.88	175.25

FT-IR Spectroscopy

The GBA of urea carbonyl and amide carbonyl (C=O) stretching frequencies were appeared at 1715cm^{-1} and 1618cm^{-1} respectively. And the stretching bands at 3315cm^{-1} and 3367cm^{-1} belongs urea and amide of N-H functional groups. These functional groups stretching bands showed significant changes in their vibrational mode regions while forming a salt. Almost all GBA salts were displayed blue shift in their stretching of N-H and C=O vibrational frequencies due to the salt formation. (All frequency changes are listed in Table 5 and spectra shown in Figure S1 and Table S2). Related to this, other functional group vibrational modes are also red/ blue shifted.

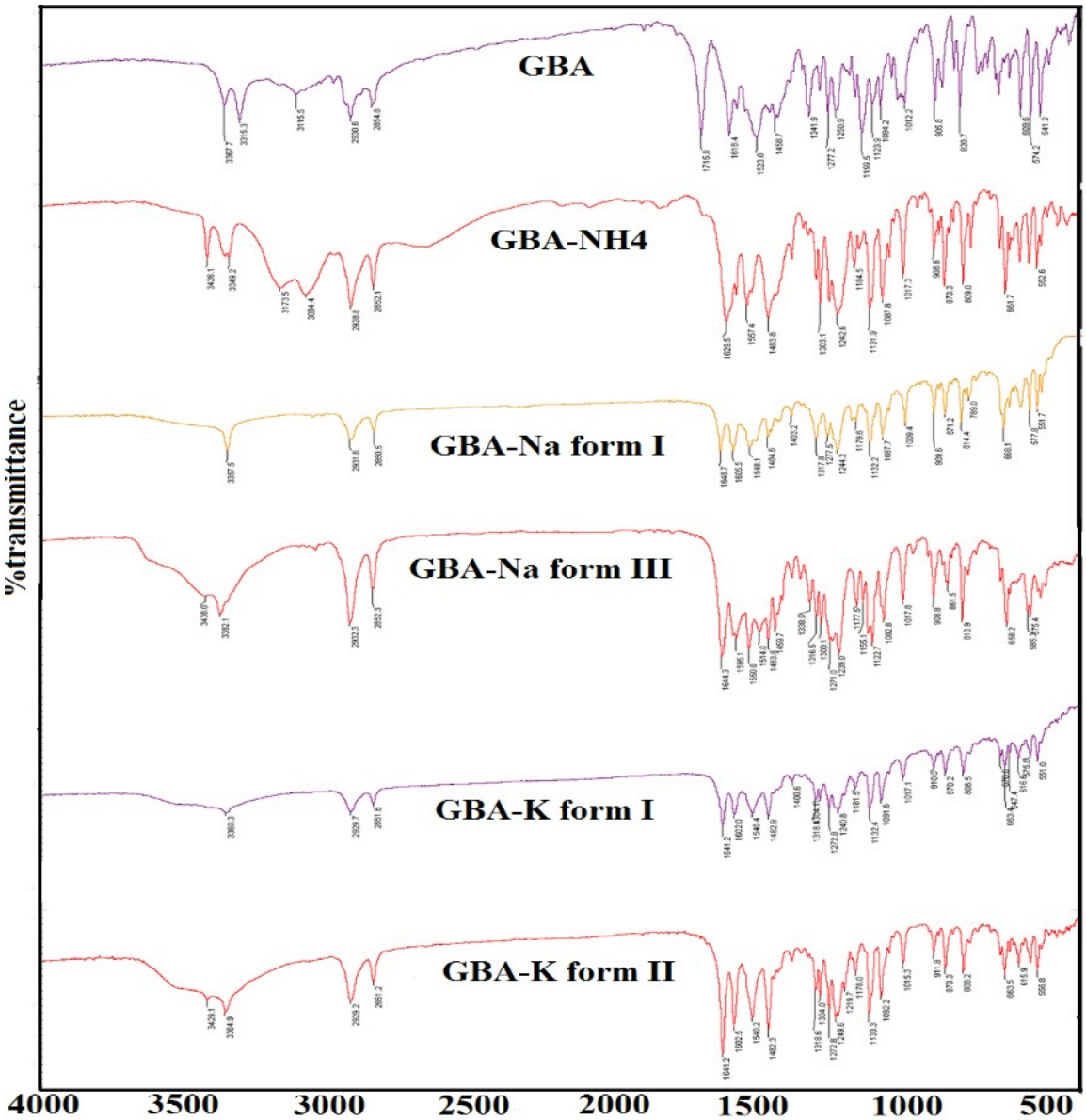


Figure S1 FT-IR spectra of GBA salts.

Table S2 IR stretching frequencies of GBA urea (NHCONH) and amide (CONH) functional groups in the solid state.

S.No	Solid form	NHCONH ($\nu_{C=O}$ in cm^{-1})	CONH ($\nu_{C=O}$ in cm^{-1})	NHCONH/CONH/H_2O ($\nu_{NH/OH}$ in cm^{-1})
1	GBA	1715	1618	3315, 3367
2	GBA-Na form I	1648	1605	3357
3	GBA-Na form III	1644	1595	3382, 3438
4	GBA-K form I	1641	1602	3360
5	GBA-K form II	1641	1602	3364, 3429
6	GBA-NH_4	1629	1598	3349, 3426

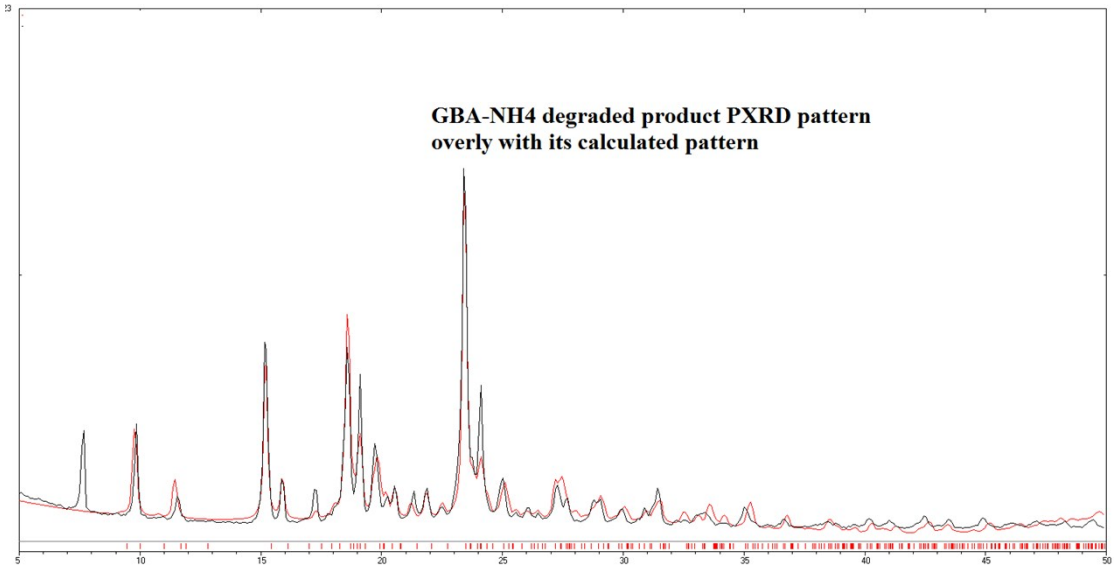


Figure S2 GBA-NH4 melted compound shown degrade into5-chloro-2-methoxy-N-(2-(4-sulfamoylphenyl)ethyl) benzamide and its calculated (red)and experimental PXRD (black) shows good match.

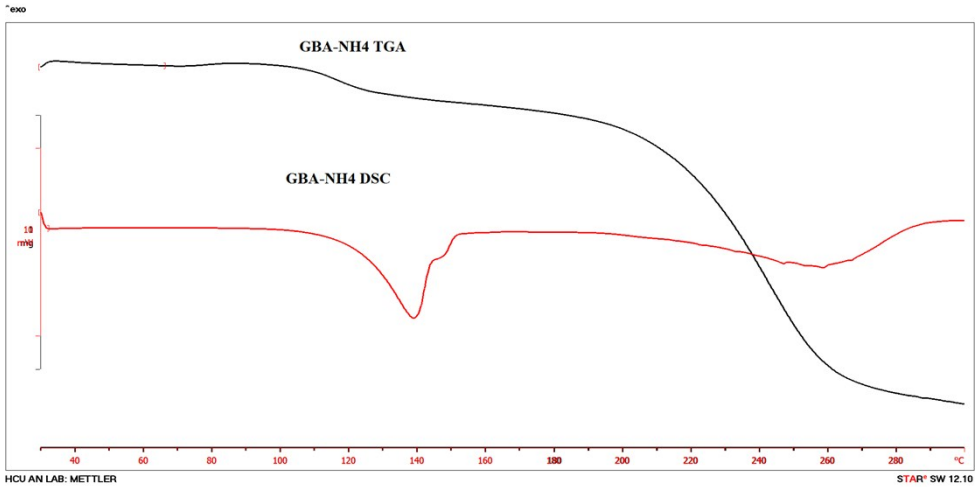


Figure S3 DSC (red) and TGA (black) of GBA-NH_4.

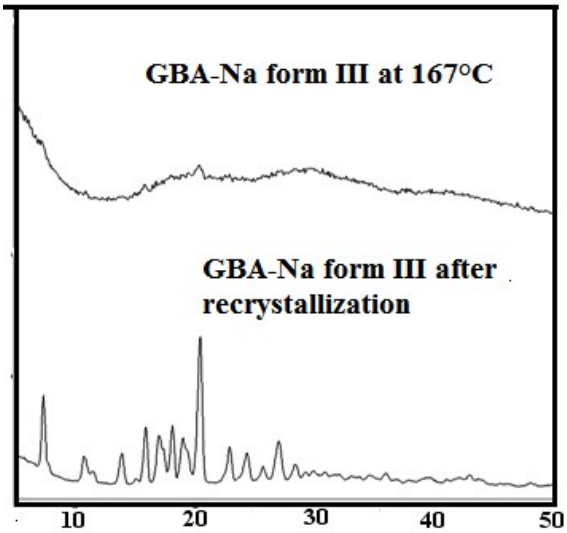


Figure S4 GBA-Na form III transforms to form I via amorphization.

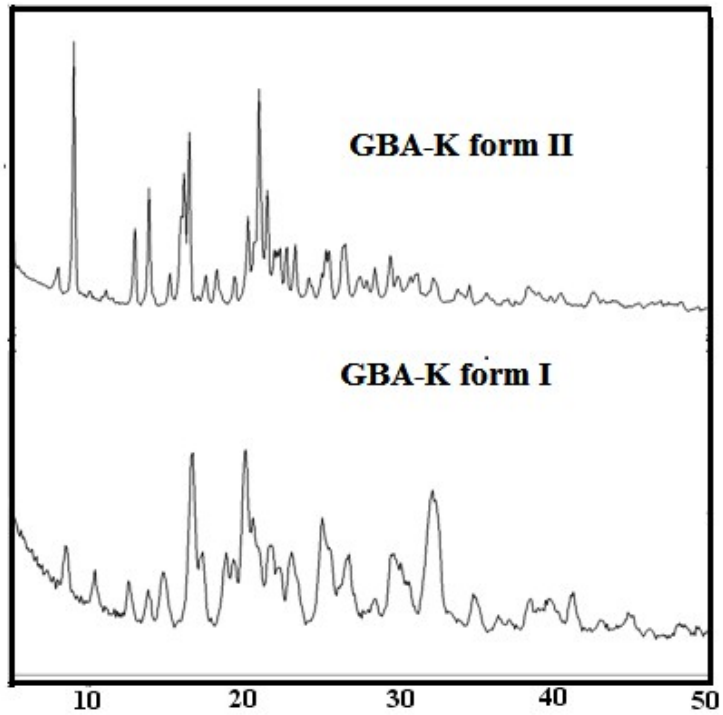


Figure S5 GBA-K form II transformation to form I upon heating at 85°C.

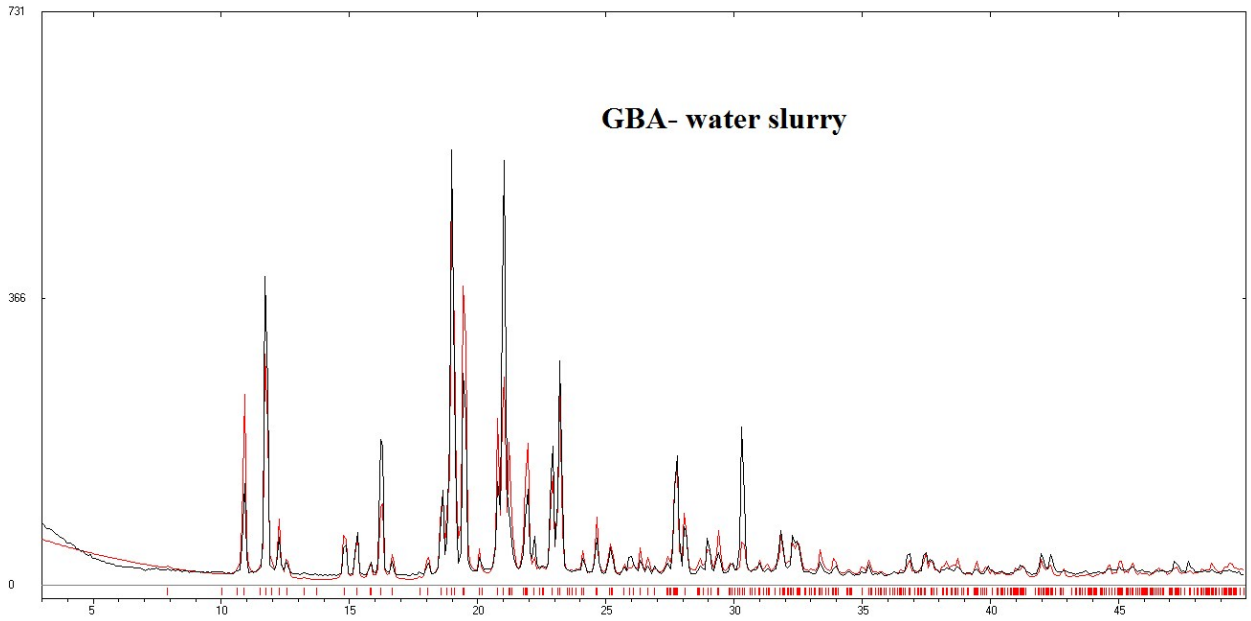


Figure S6a PXRD plot of GBA at the end of equilibrium solubility experiment in water medium. Red color trace is the calculated pattern from the X-ray structure and black profile is the experimental powder XRD peaks shows good match.

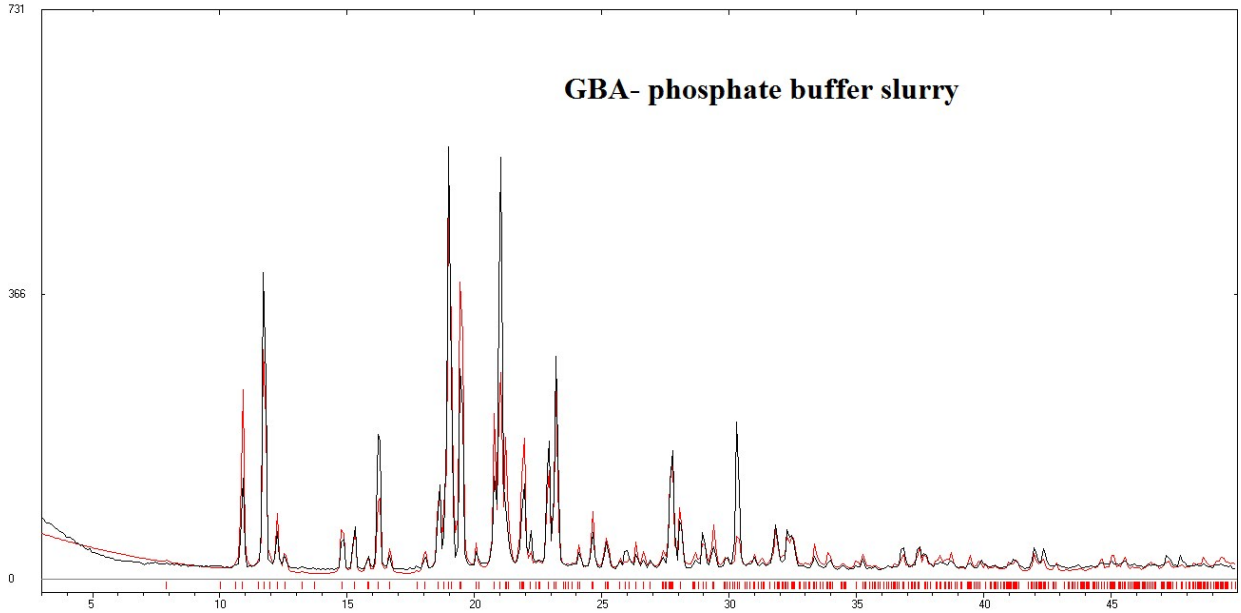


Figure S6b PXRD plot of GBA at the end of equilibrium solubility experiment in pH 7 phosphate buffer medium. Red color trace is the calculated pattern from the X-ray structure and black profile is the experimental powder XRD peaks shows good match.

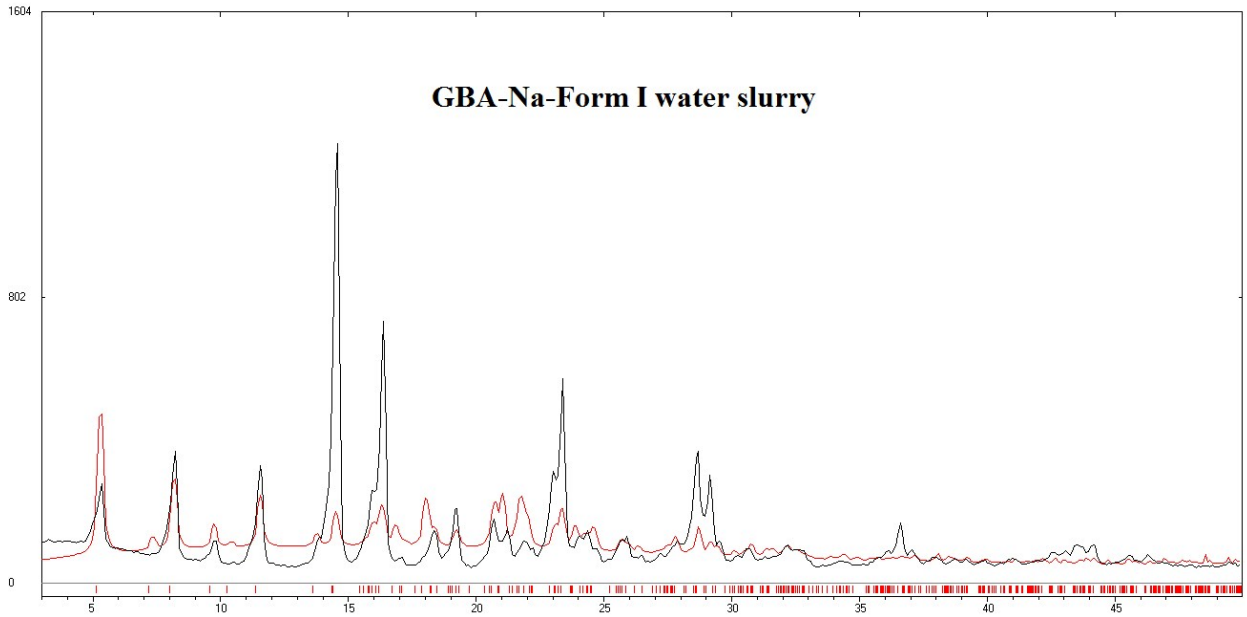


Figure S6c PXRD plot of GBA-Na form I at the end of equilibrium solubility experiment in water medium. Red color trace is the calculated pattern from the X-ray structure of GBA-Na-form III and black profile is the experimental powder XRD peaks shows good match. It indicates that during slurry experiment form I is converted into Form III (hydrate).

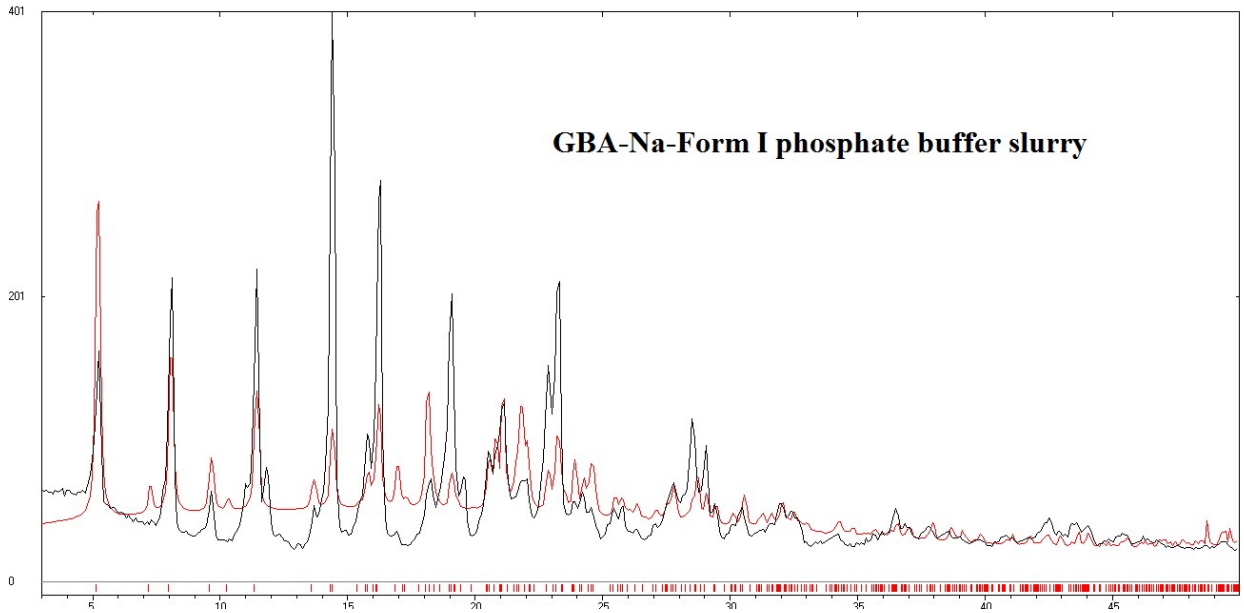


Figure S6d PXRD plot of GBA-Na form I at the end of equilibrium solubility experiment in phosphate buffer medium. Red color trace is the calculated pattern from the X-ray structure of GBA-Na-form III and black profile is the experimental powder XRD peaks shows good match. It indicates that during slurry experiment form I is converted into Form III (hydrate).

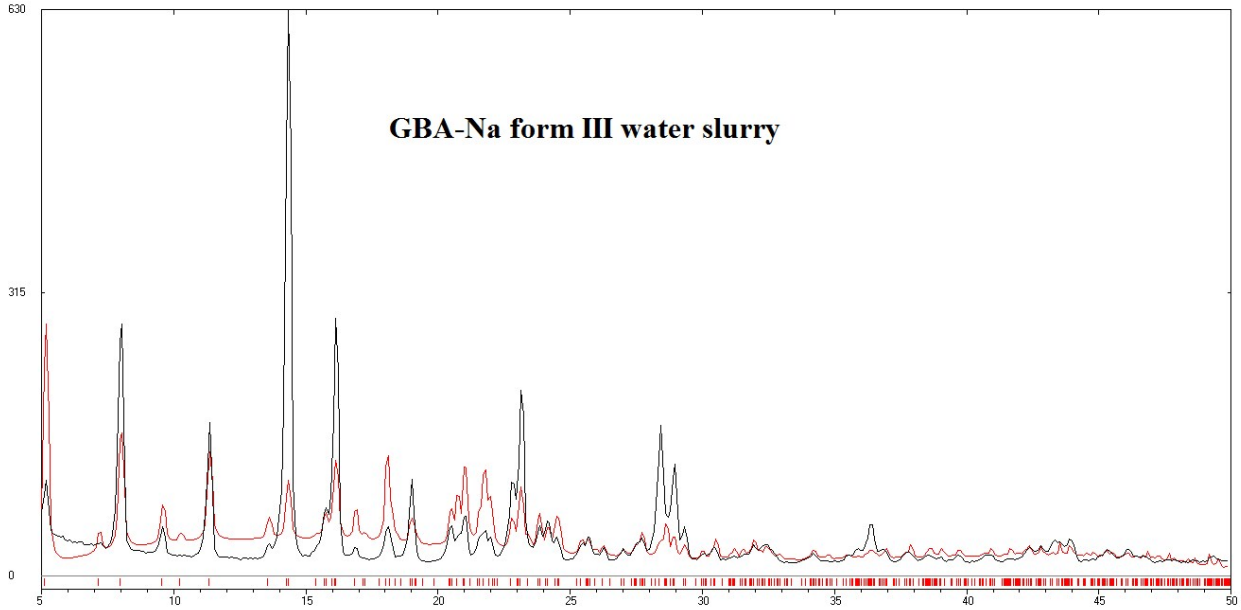


Figure S6e PXRD plot of GBA-Na form III at the end of equilibrium solubility experiment in water medium. Red color trace is the calculated pattern from the X-ray structure and black profile is the experimental powder XRD peaks shows good match.

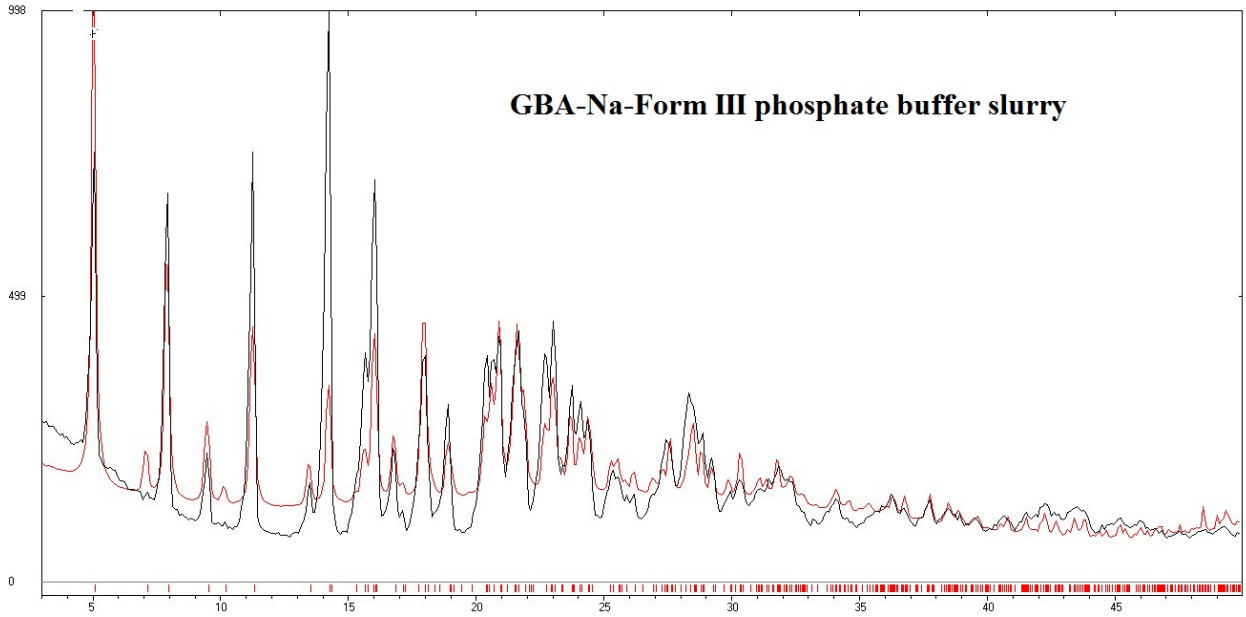


Figure S6f PXRD plot of GBA-Na form III at the end of equilibrium solubility experiment in phosphate buffer medium. Red color trace is the calculated pattern from the X-ray structure and black profile is the experimental powder XRD peaks shows good match.

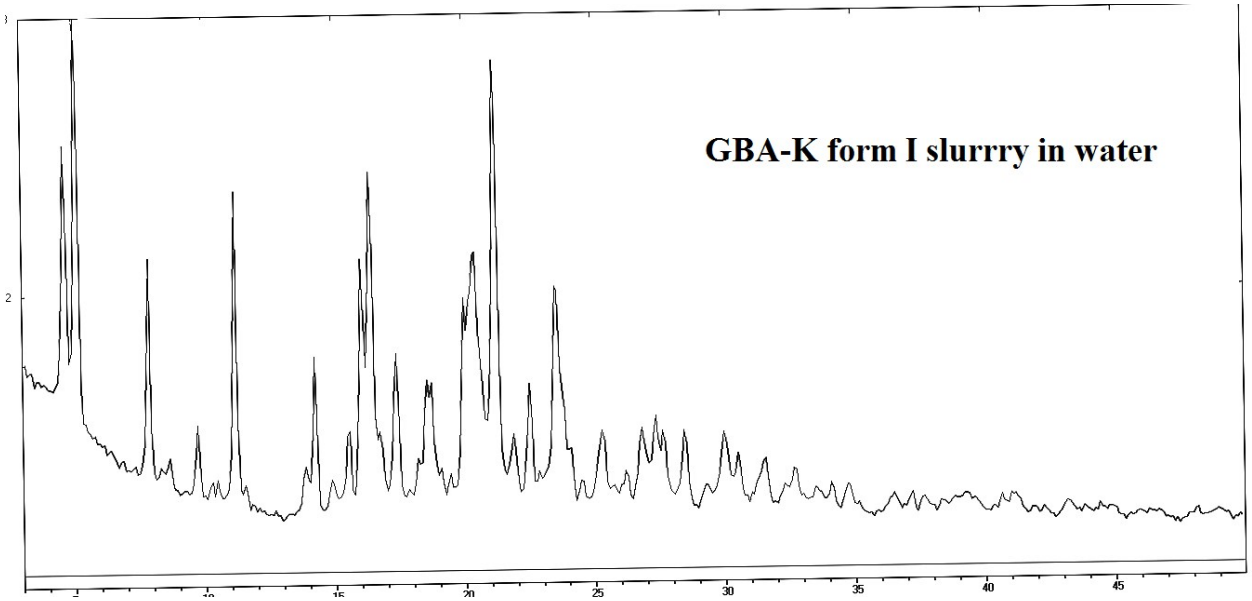


Figure S6g PXRD plot of GBA-K form I at the end of equilibrium solubility experiment in water medium and it is transformed into its hydrate (form II).

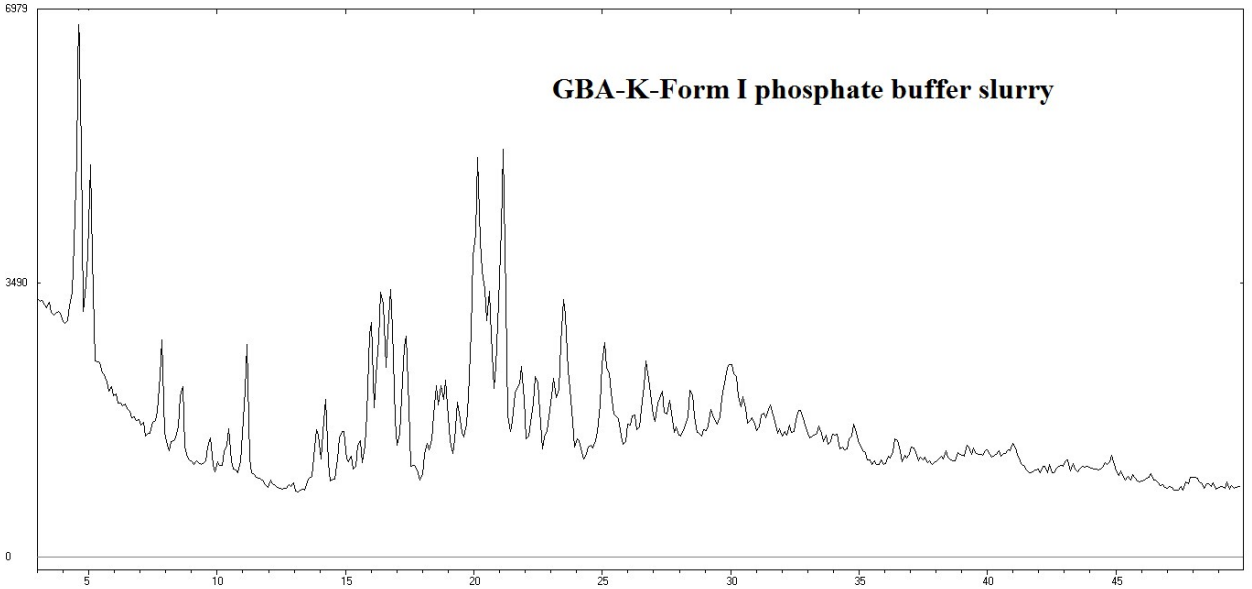


Figure S6h PXRD plot of GBA-K form I at the end of equilibrium solubility experiment in phosphate buffer medium and it is transformed into its hydrate (form II).

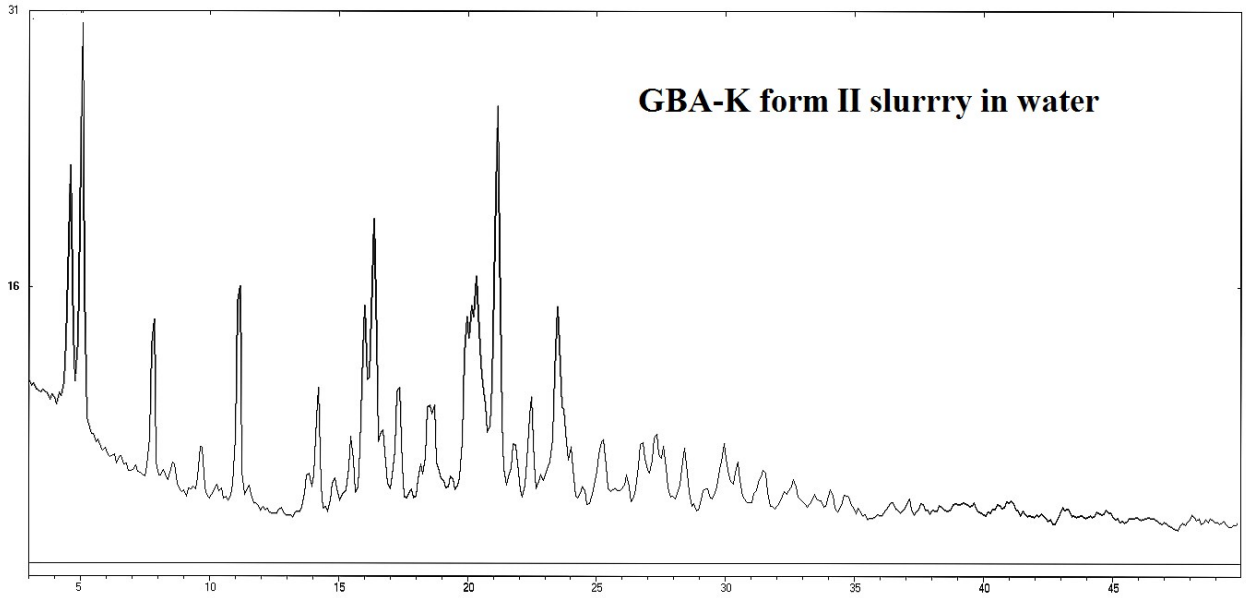


Figure S6i PXRD plot of GBA-K form II at the end of equilibrium solubility experiment in water medium and it matches with starting material indicates is stable during slurry experiment.

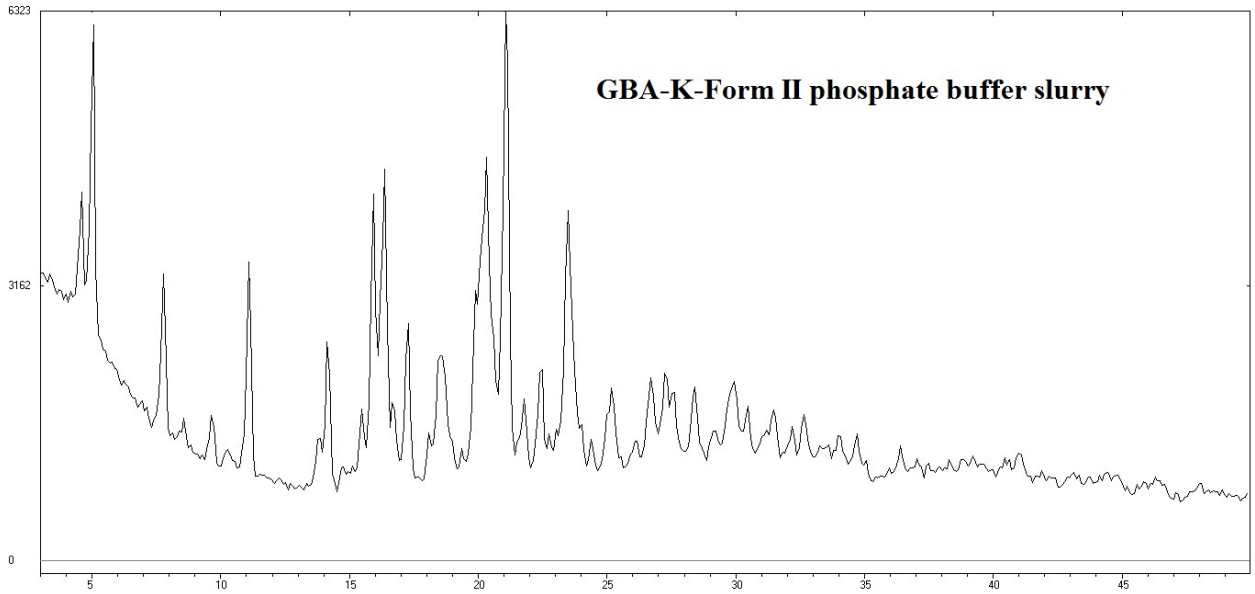


Figure S6j PXRD plot of GBA-K form II at the end of equilibrium solubility experiment in water medium and it matches with starting material indicates is stable during slurry experiment.

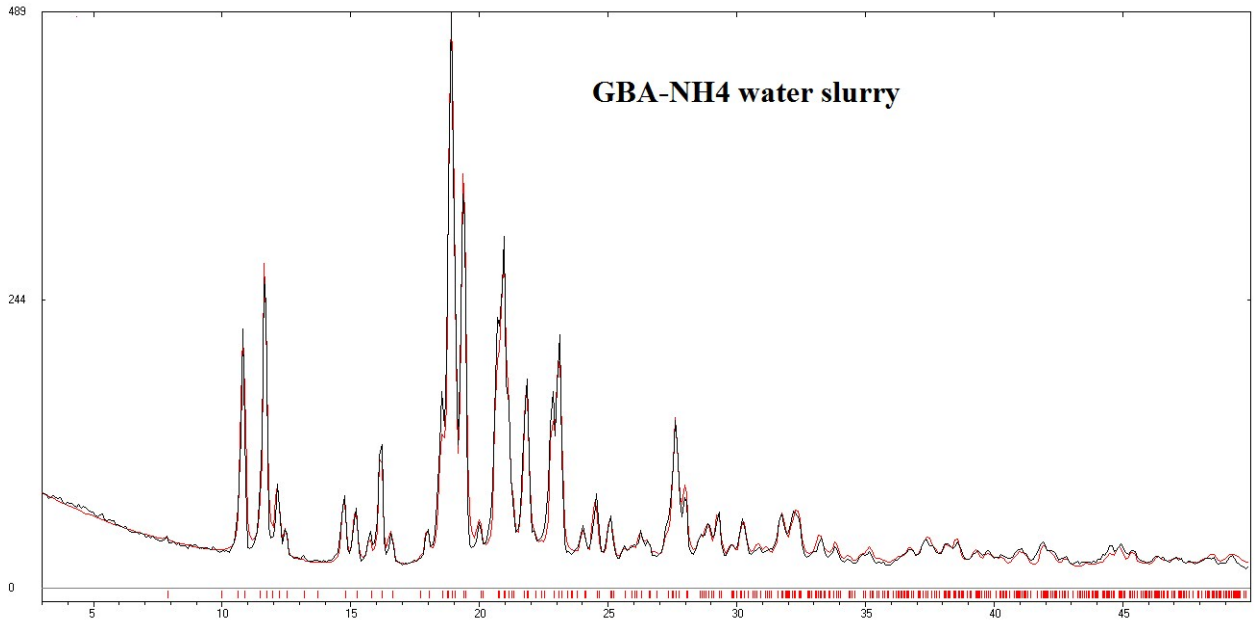


Figure S6k PXRD plot of GBA-NH_4 at the end of equilibrium solubility experiment in phosphate buffer medium. Red color trace is the calculated pattern from the X-ray structure of GBA and black profile is the experimental powder XRD peaks shows good match. It indicates that during slurry experiment salt disproportionation is occurred.

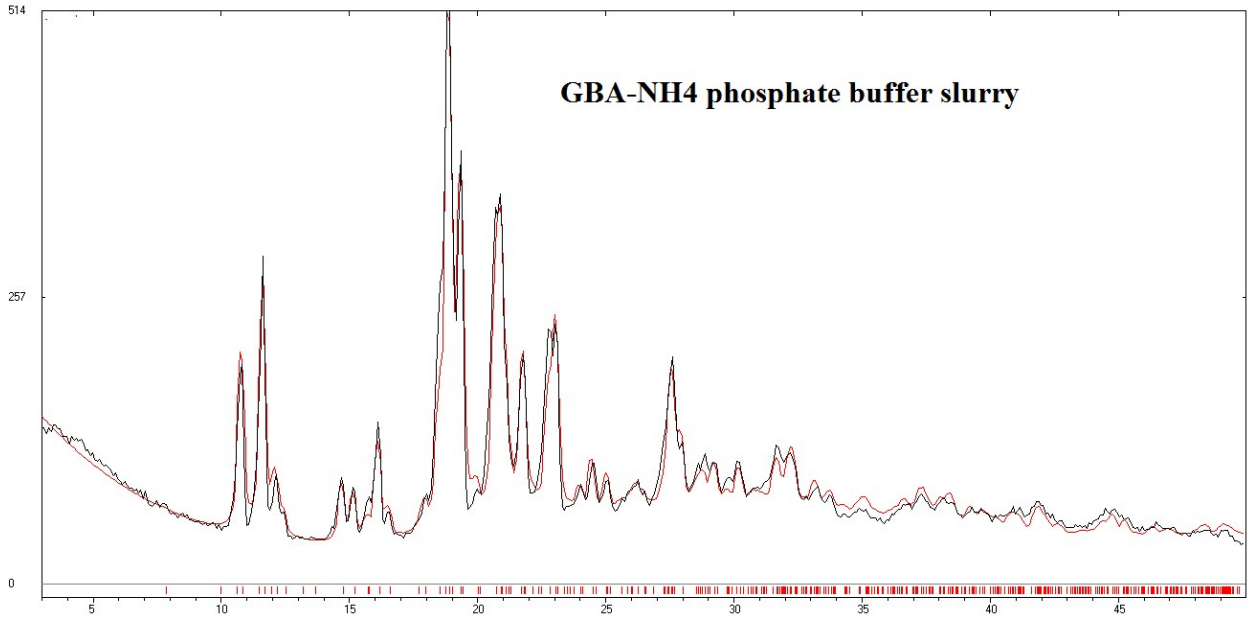


Figure S6l PXRD plot of GBA-NH_4 at the end of equilibrium solubility experiment in phosphate buffer medium. Red color trace is the calculated pattern from the X-ray structure of GBA and black profile is the experimental powder XRD peaks shows good match. It indicates that during slurry experiment salt disproportionation is occurred.

Figure 6a-l Powder XRD plots of Glibenclamide salts.

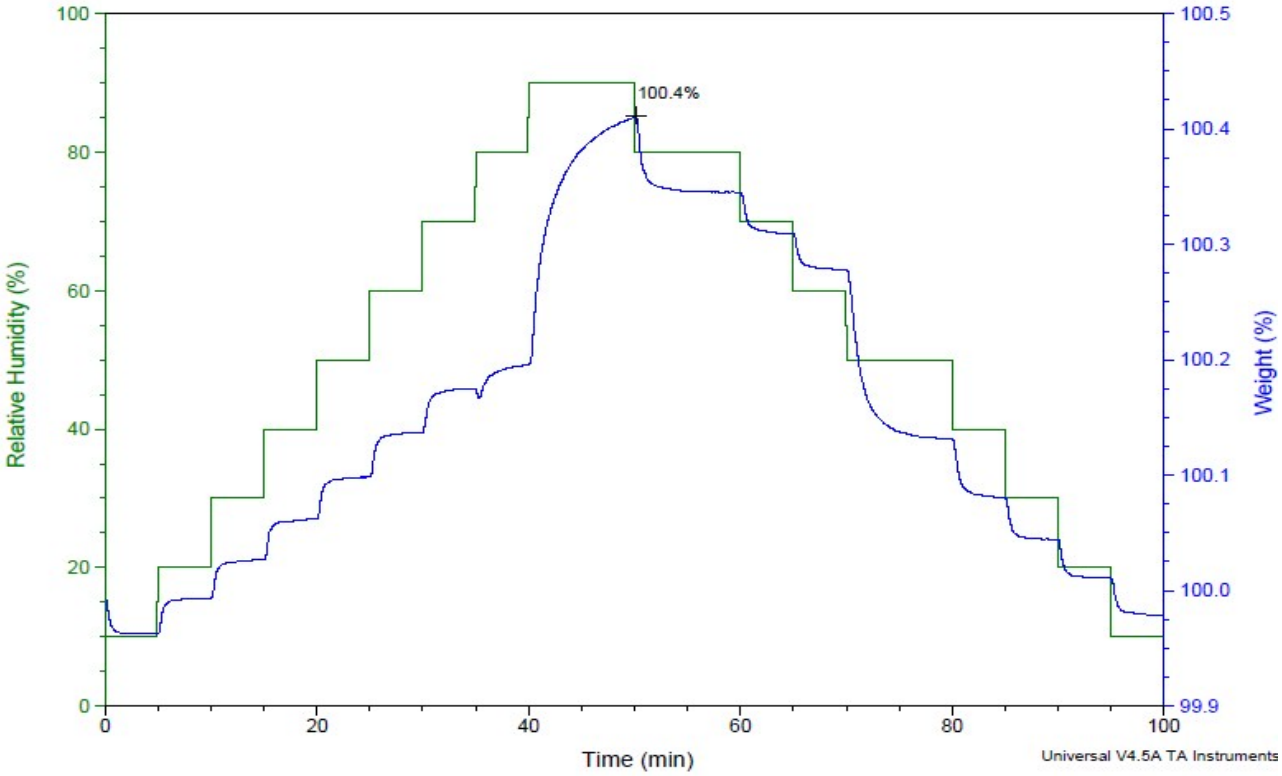


Figure S7a GBA-Na form I (adsorbed water, 0.4%).

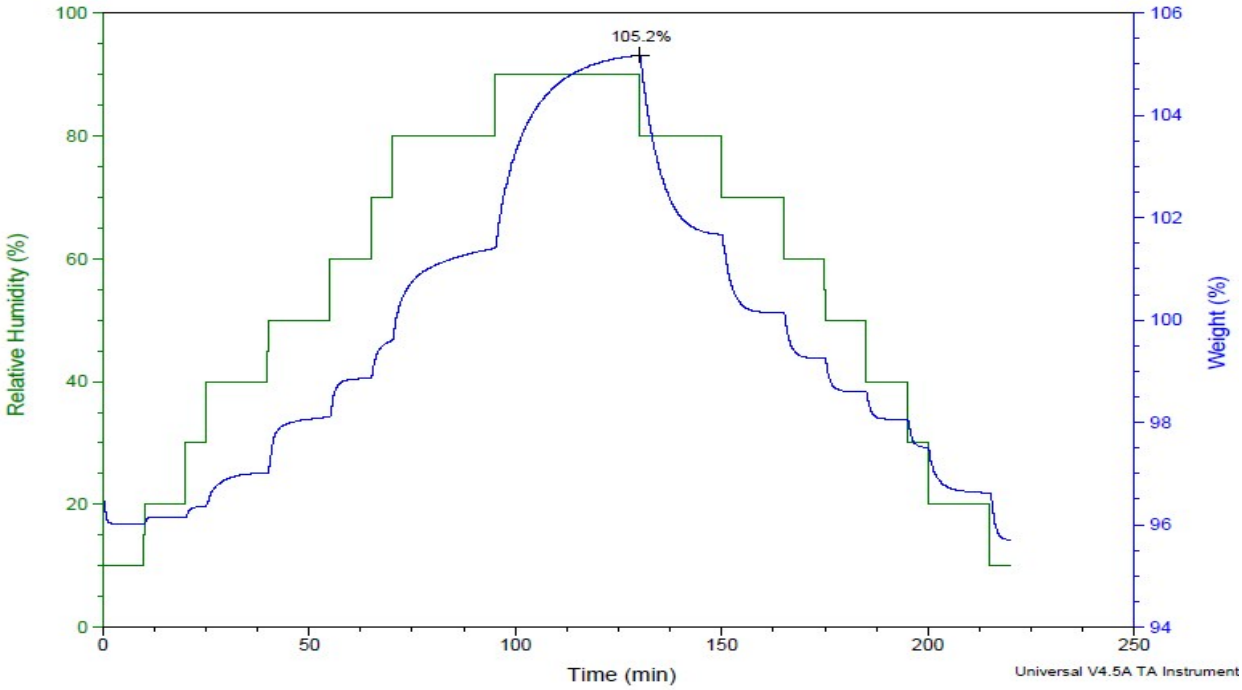


Figure S7b GBA-Na form III (adsorbed water, 5.2%).

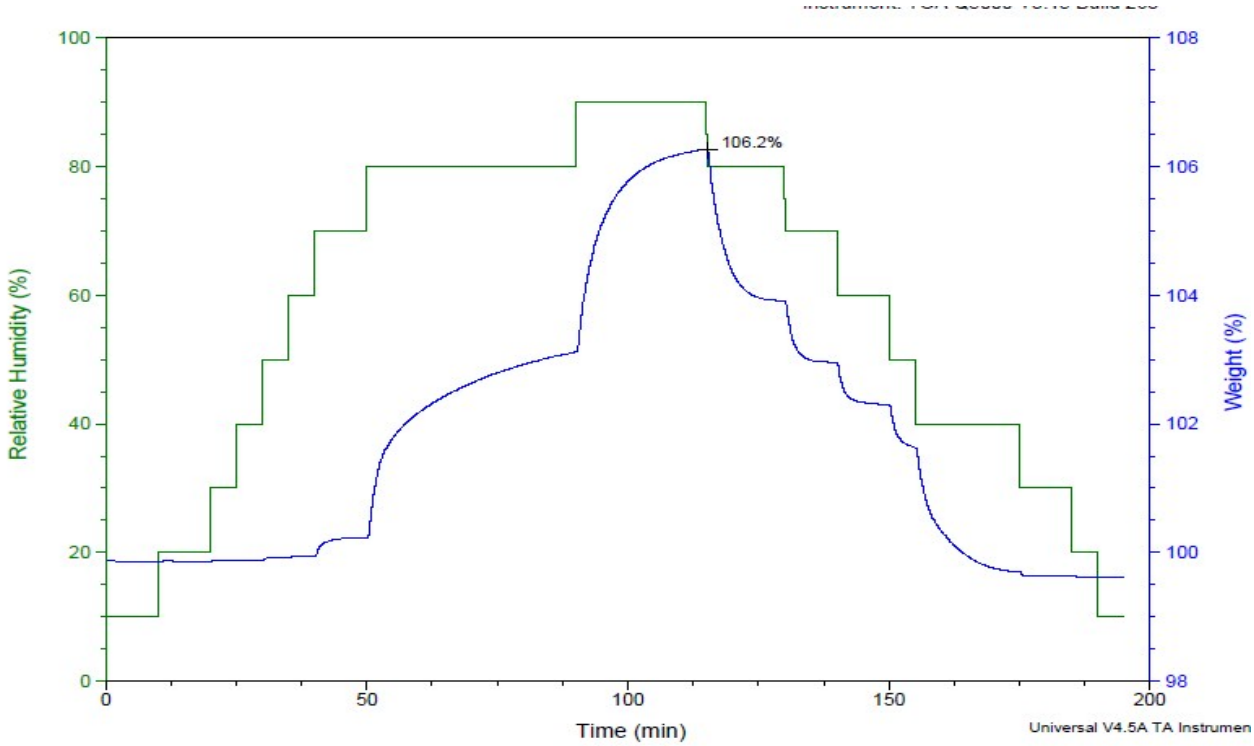


Figure S7c GBA-K form I (adsorbed water, 6.2%).

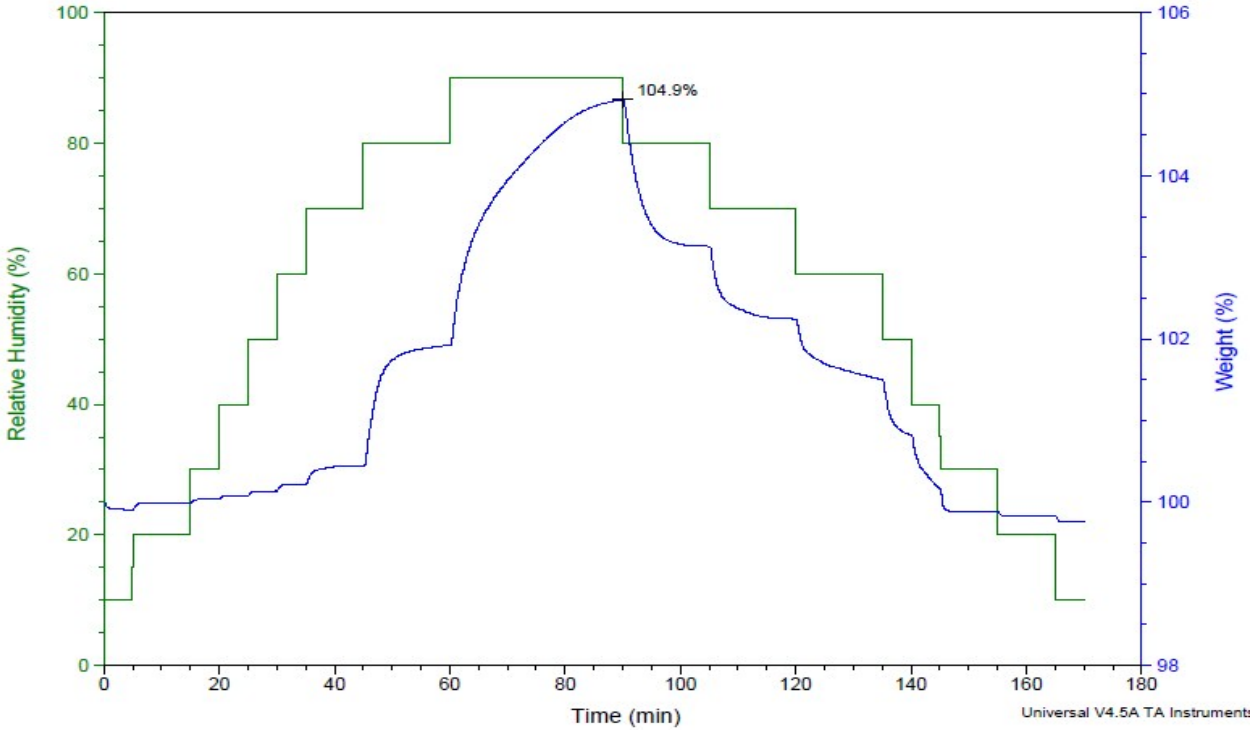


Figure S7d GBA-K form II (adsorbed water, 4.9%).

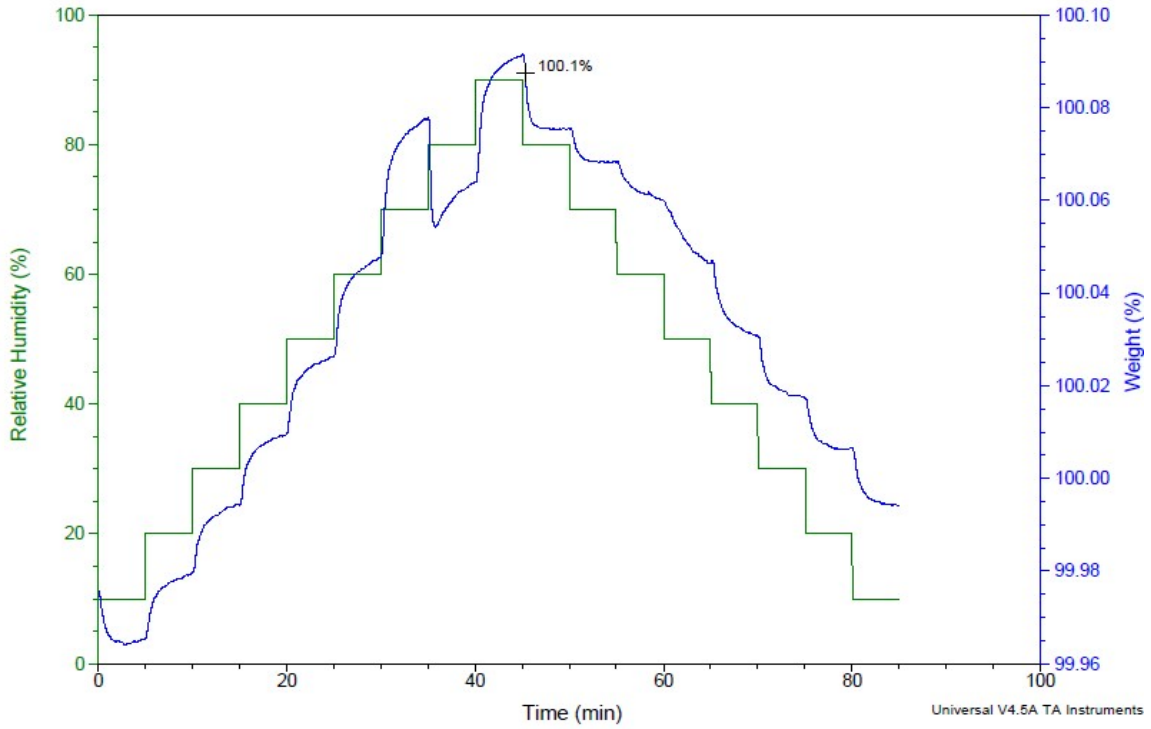


Figure S7e GBA-NH_4 (adsorbed water, 0.1%).

Figure S7a-e DVS plots of GBA-Na, GBA-K and GBA-NH_4 forms.

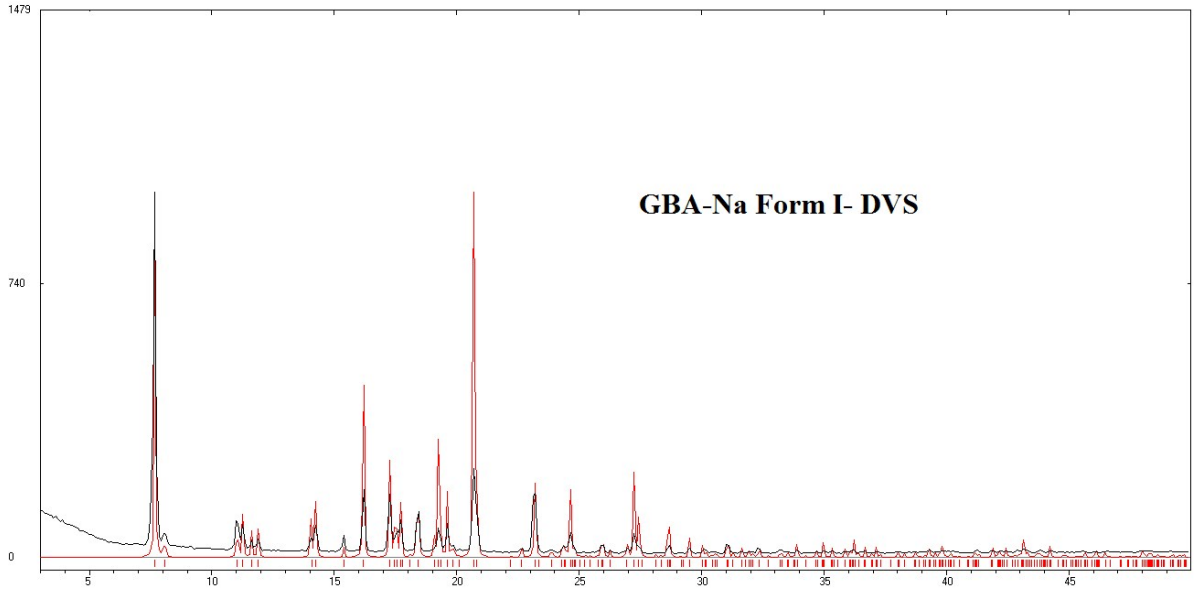


Figure S8a PXRD plot of GBA-Na form I recorded at the end of DVS analysis. Red color profile is calculated powder pattern from the X-ray crystal structure and black pattern is experimental powder XRD peaks shows good match.

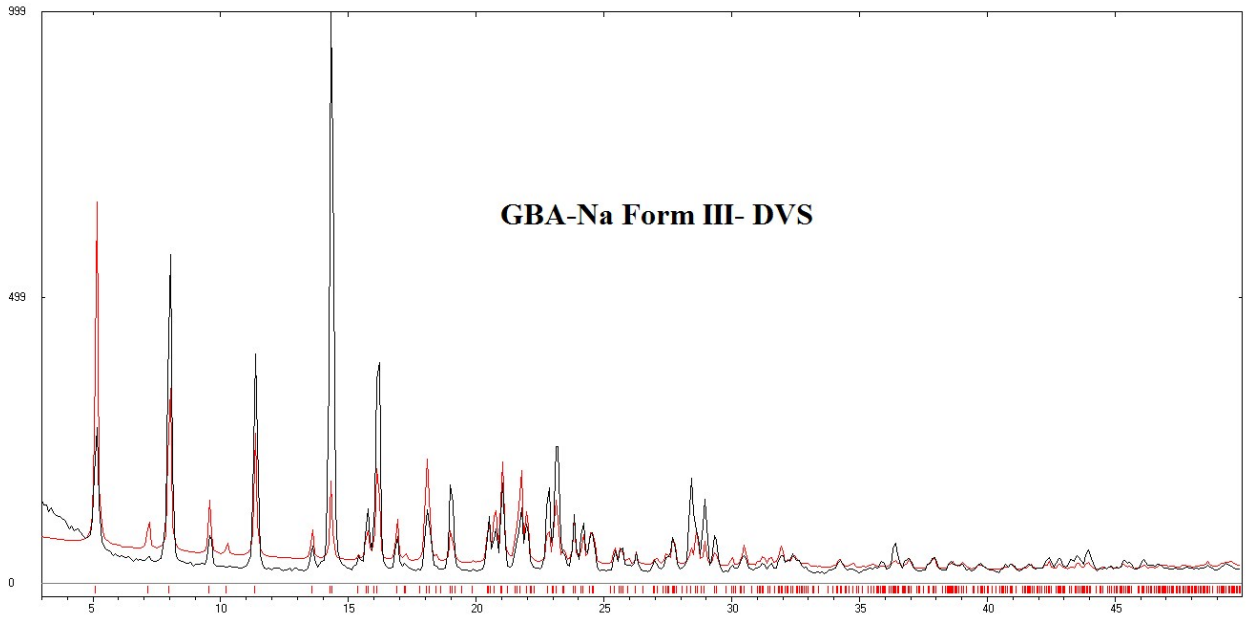


Figure S8b PXRD plot of GBA-Na form III recorded at the end of DVS analysis. Red color profile is calculated powder pattern from the X-ray crystal structure and black pattern is experimental powder XRD peaks shows good match.

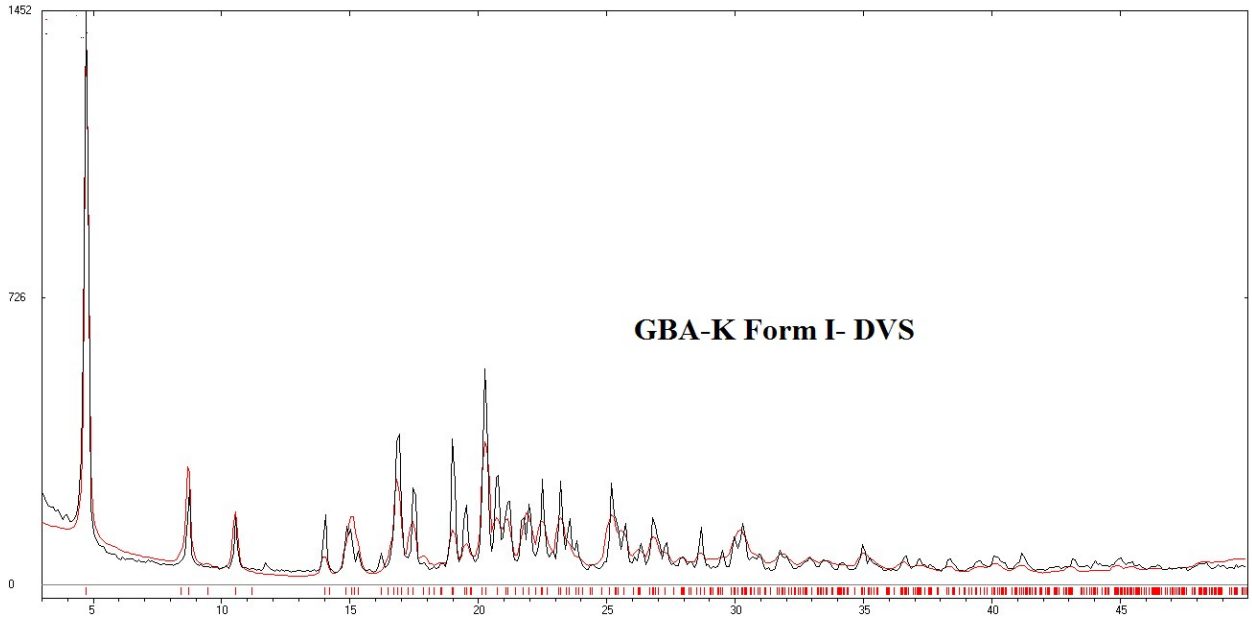


Figure S8c PXRD plot of GBA-K form I recorded at the end of DVS analysis. Red color profile is calculated powder pattern from the X-ray crystal structure and black pattern is experimental powder XRD peaks shows good match.

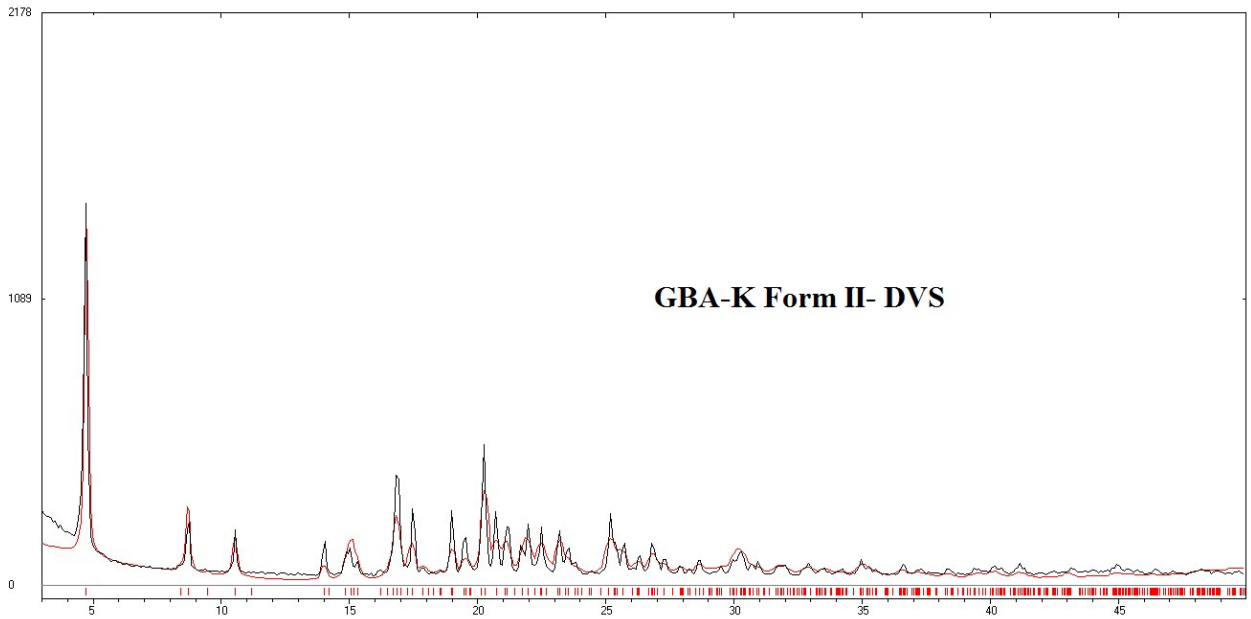


Figure S8d PXRD plot of GBA-K form II recorded at the end of DVS analysis. Red color profile is calculated powder pattern from the X-ray crystal structure of GBA-K form I and black pattern is experimental powder XRD peaks shows good match. It indicates that GBA-K form II is converted into form I during DVS experiment.

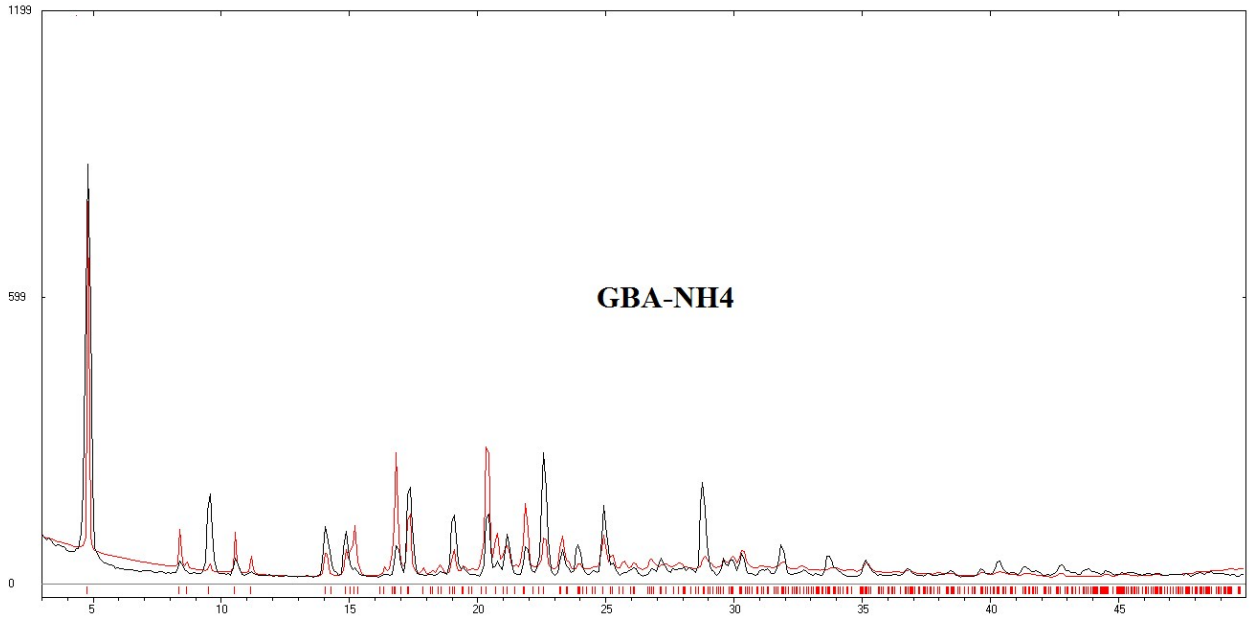


Figure S8e PXRD plot of GBA-NH_4 recorded at the end of DVS analysis. Red color profile is calculated powder pattern from the X-ray crystal structure and black pattern is experimental powder XRD peaks shows good match.

Figure S8a-e PXRD plots of GBA salt forms recorded at the end of DVS analysis.