Polymorphs of daidzein and intermolecular interaction effect on solution crystallization

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Contents of Supporting Information

Figure S1

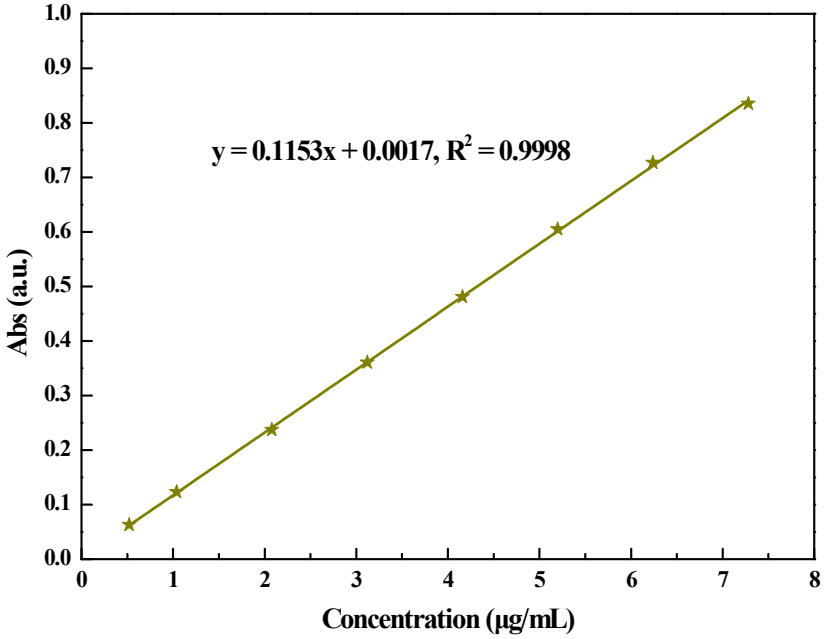


Figure S1. UV standard curve.

Figure S2

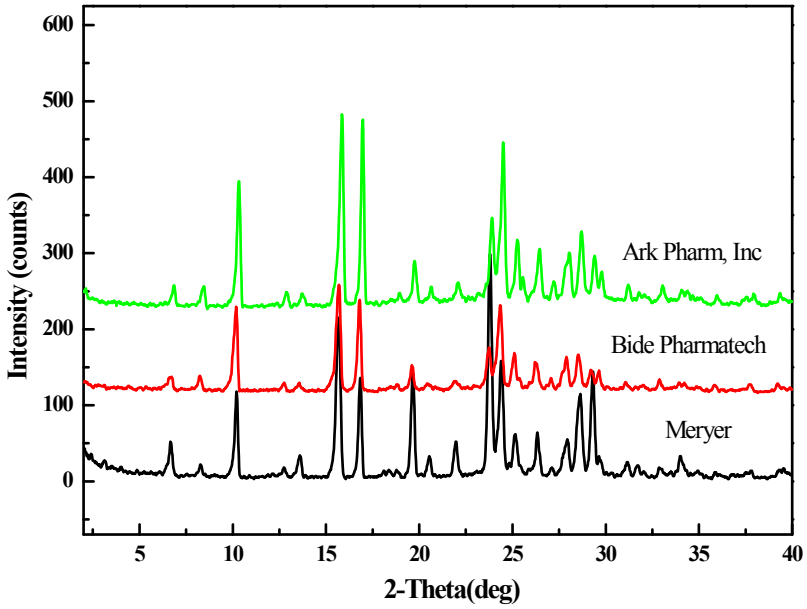
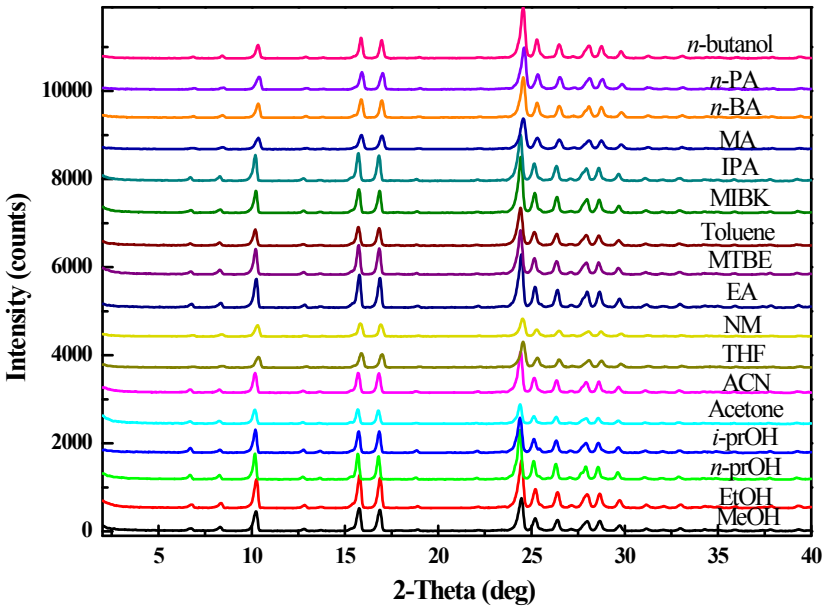
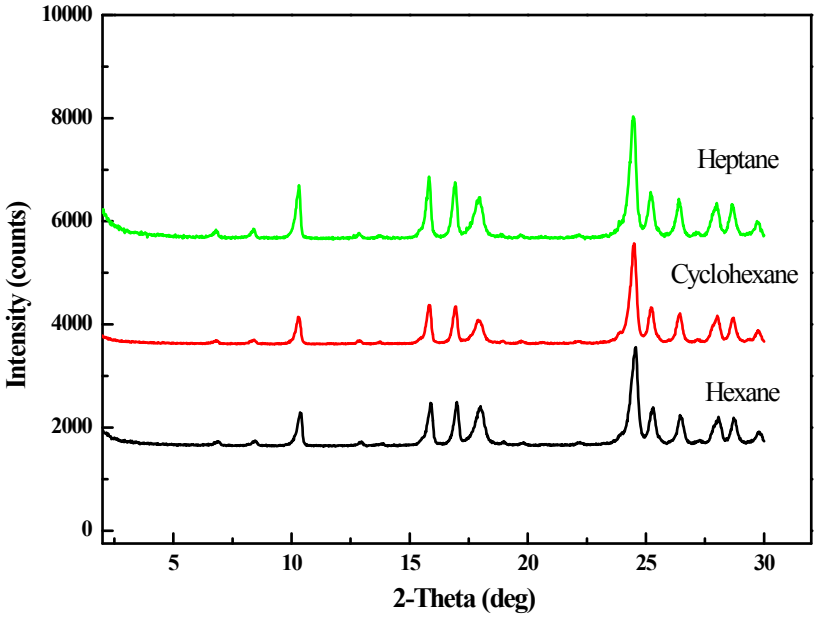


Figure S2. PXRD patterns of form I from different batches.

Figure S3


Intensity (counts)
2-Theta (deg)
n-butanol
n-PA
n-BA
MA
IPA
MIBK
Toluene
MTBE
EA
NM
THF
ACN
Acetone
i-prOH
n-prOH
EtOH
MeOH

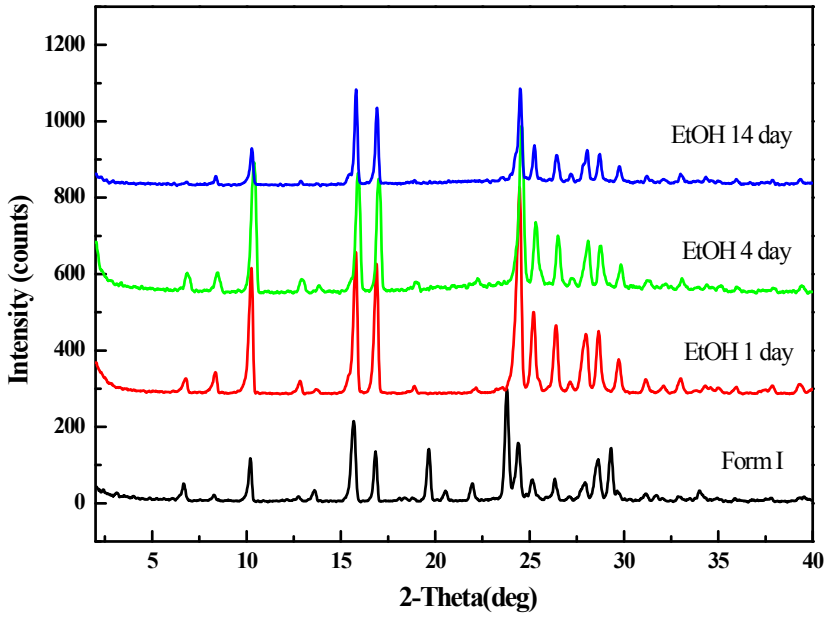
(a)


Intensity (counts)
2-Theta (deg)
Heptane
Cyclohexane
Hexane

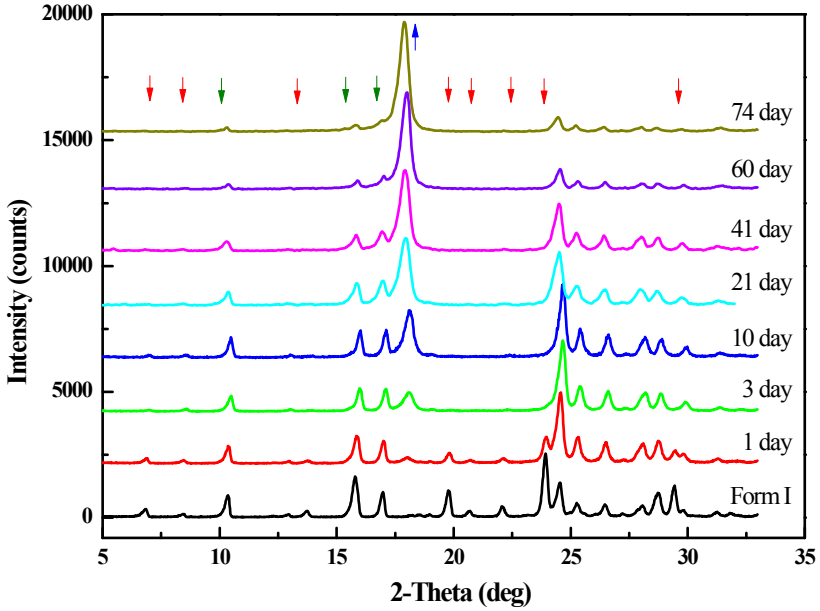
(b)

Figure S3. (a) Form II is preferred when slurry in most solvents, and (b) form III is preferred in hexane, cyclohexane and heptane.

Figure S4



(a)



(b)

Figure S4. Different forms need different time for transformation, (a) form II prepared in EtOH, (b) form III prepared in Hex (red arrow means peak disappeared, green arrow means peak decreased and blue arrow means new peak appeared).

Table S1

Table S1. Crystallographic data of DAID form II and DMF solvate.

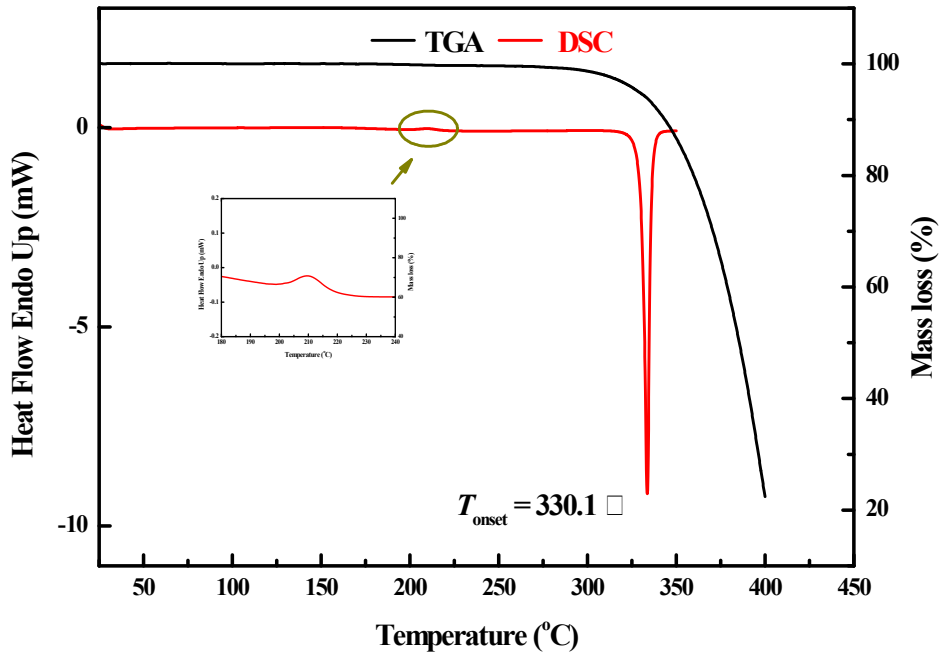
	Form I	DMF solvate
Formula	$C_{15}H_{10}O_4$	$C_{15}H_{10}O_4$▪C_3H_7NO
Crystal system	Orthorhombic	Monoclinic
Space group	*P* 2_1 2_1 2_1	*P* 2_1 /n
Temperature (K)	293	293
a (Å)	3.8600 (8)	6.260 (4)
b (Å)	11.430 (2)	20.454 (12)
c (Å)	25.629 (5)	12.386 (8)
β (°)	90	97.146 (10)
Cell volume ($Å^3$)	1130.8 (4)	1573.8 (16)
Calc.density (g/cm^3)	1.493	1.382
Z	4	4
R_{int}	0.0699	0.0235
R_1 (I>2sigma (I))	0.0667	0.0497
wR_2	0.1053	0.1214
GOF (S)	1.103	1.069
CCDC	1561888	1479812

Table S2

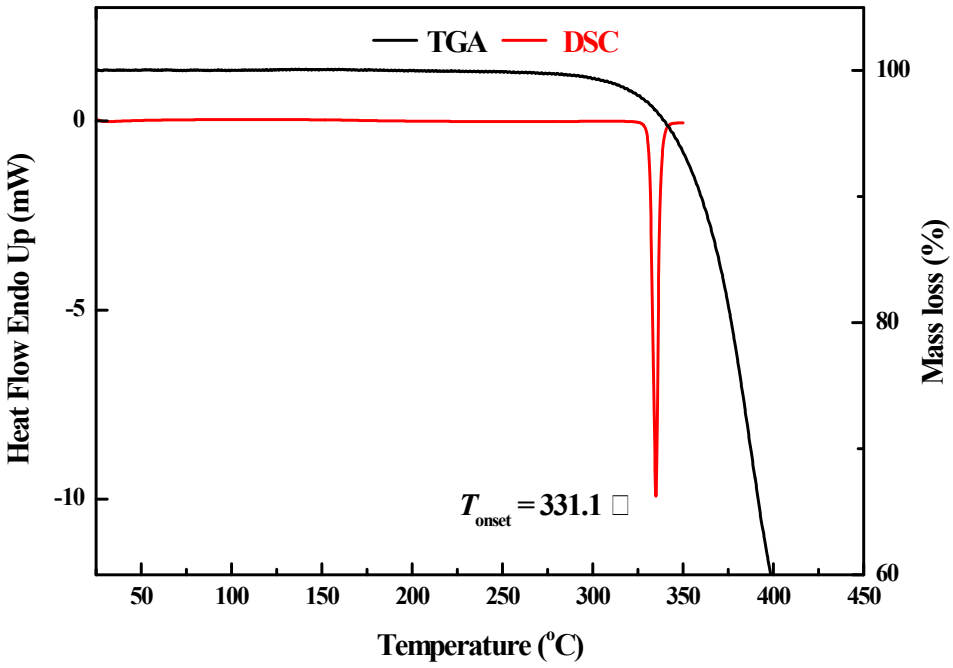
Table S2. The hydrogen bonds of DAID form II.

D–H···A	d (D–H) (Å)	d (H···A) (Å)	d (D···A) (Å)	θ (DHA) (°)	Symmetry code
O_4–H···O_2	0.82	1.88	2.701 (2)	175.9	-x, y-1/2, -z+1/2
O_3–H···O_4	0.82	1.93	2.745 (2)	173.6	-x+1/2, -y+1, z+1/2

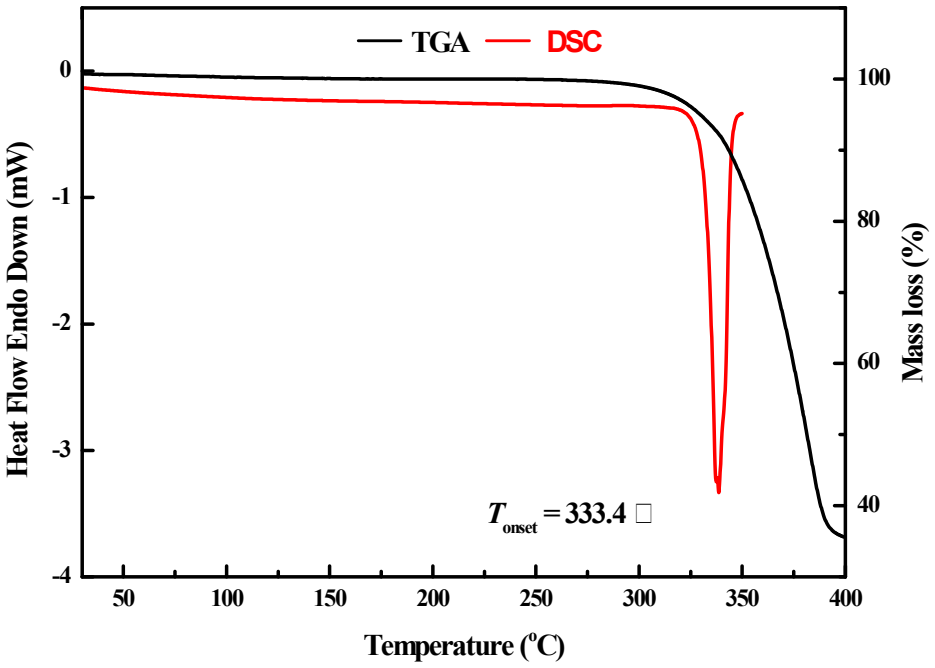
Figure S5


TGA
DSC
Heat Flow Endo Up (mW)
Mass loss (%)
Temperature (°C)
T_{onset} = 330.1 □
0
-5
-10
100
80
60
40
20
50
100
150
200
250
300
350
400
450

(a)


TGA
DSC
Heat Flow Endo Up (mW)
Mass loss (%)
Temperature (°C)
T_{onset} = 331.1 □
0
-5
-10
100
80
60
50
100
150
200
250
300
350
400
450

(b)



(c)

Figure S5. The overlaid DSC and TGA profiles of (a) form I, (b) form II and (c) form III.

Figure S6

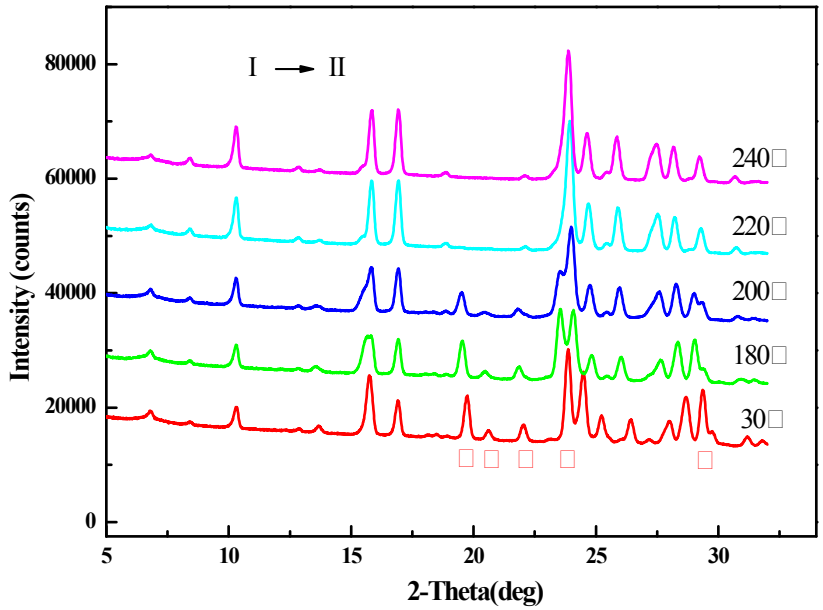
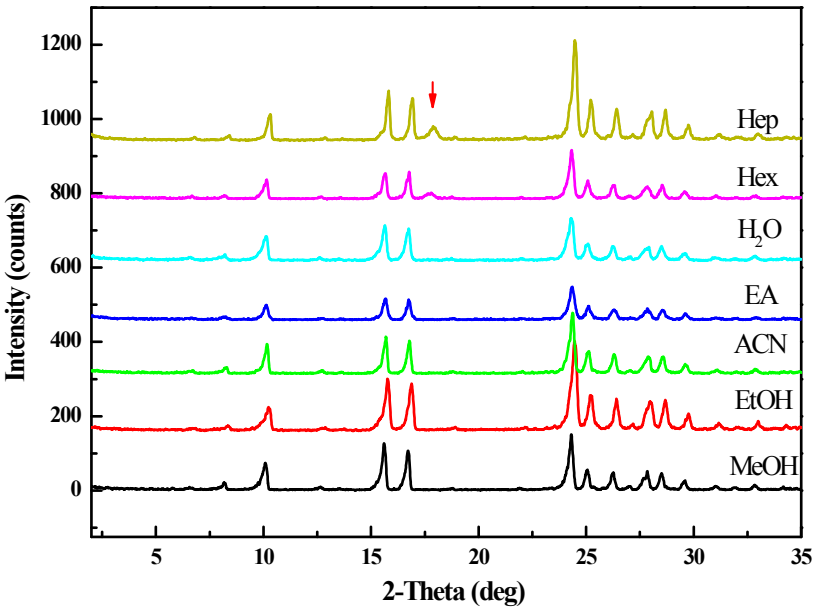
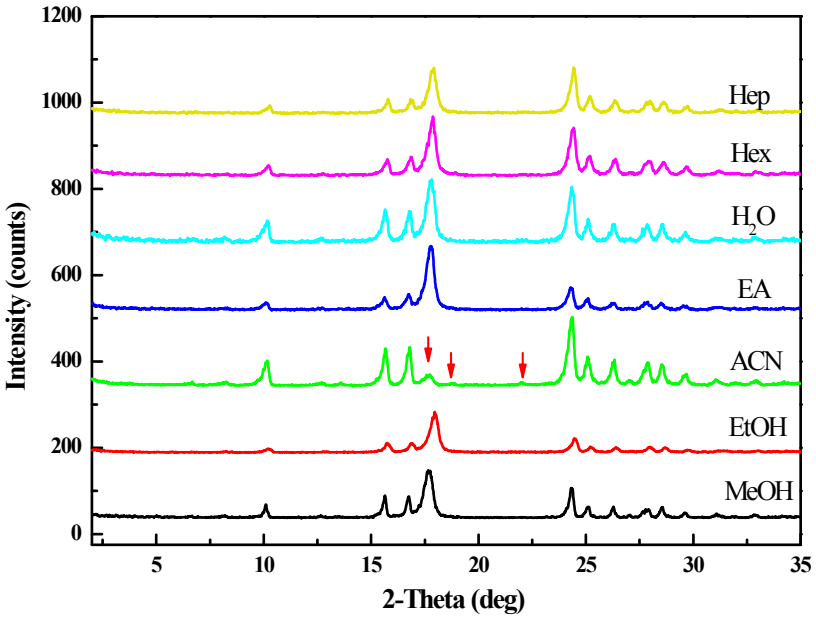


Figure S6. Form transformation process of form I was monitored by VT-PXRD.

Figure S7



(a)



(b)

Figure S7. Stability of (a) form II and (b) form III in different solvent media.

Figure S8

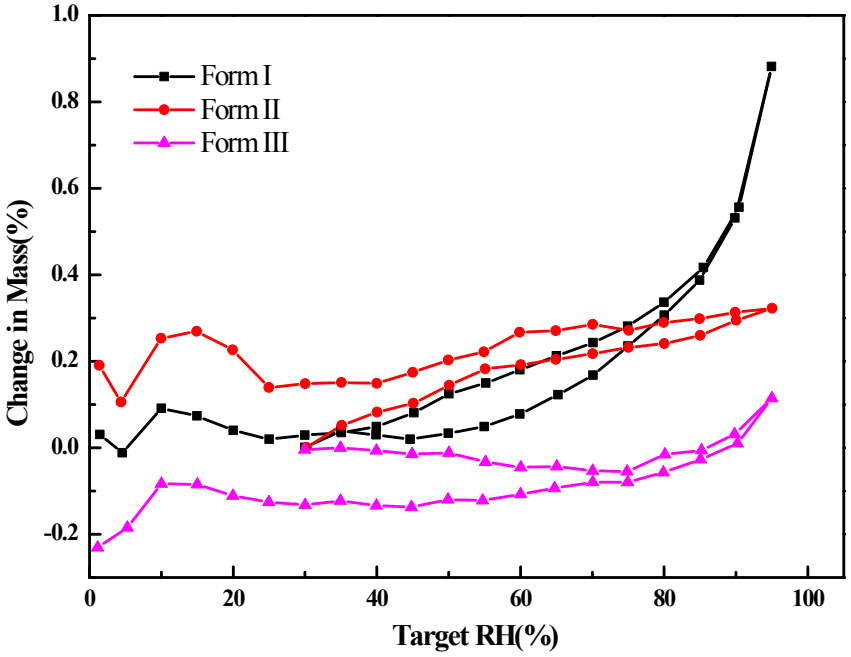
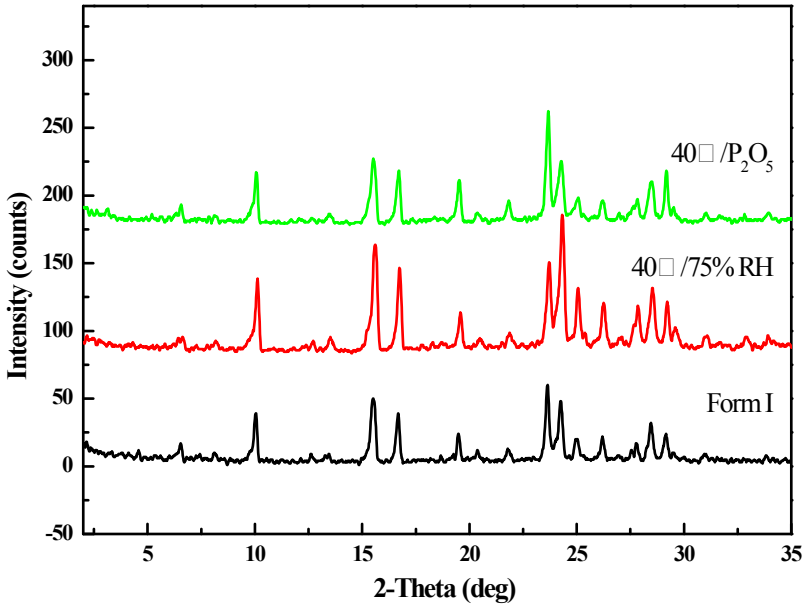
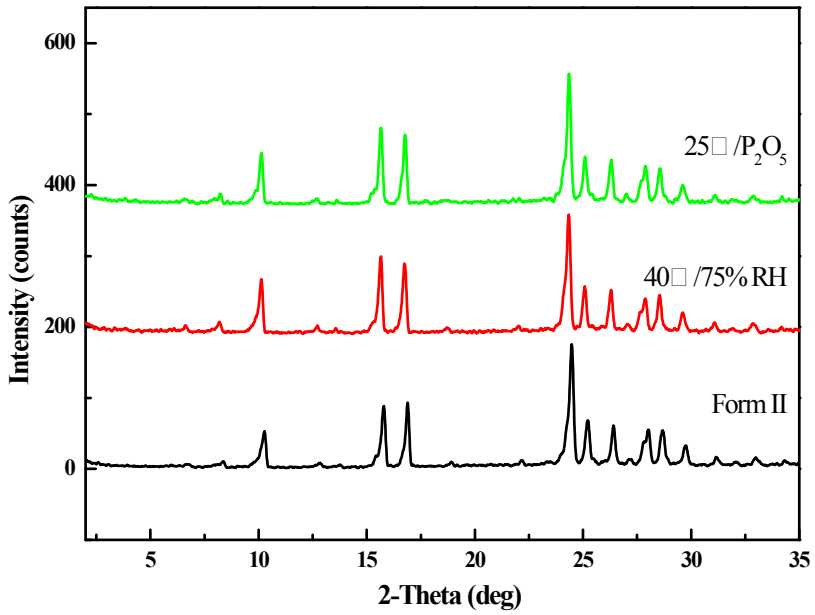


Figure S8. DVS diagrams of various solid forms of DAID.

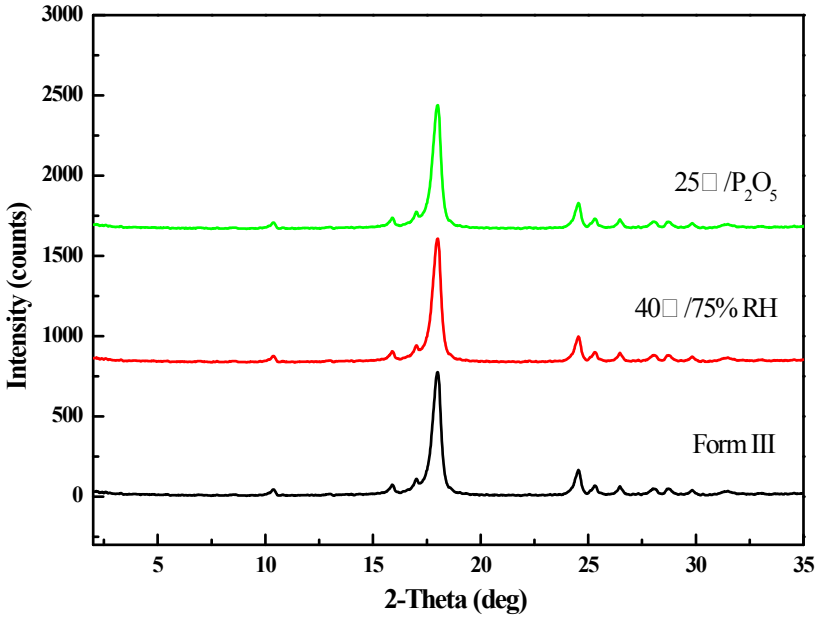
Figure S9



(a)



(b)



(c)

Figure S9. Accelerated stability of (a) form I, (b) form II and (c) form III for 8 weeks.

Table S3

Table S3. Dissolution percentage of various forms in different dissolution media after 20 min.

	I	II	III
Water (%)	46.9	81.5	4.3
pH 1.2 (%)	64.2	78.6	9.3
pH 6.8 (%)	39.4	74.7	4.1