Supporting Information

Facile Preparation Method for Inclusion Complexes between Amylose and Polytetrahydrofurans

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Figure 1. Pressure vessel for preparing amylose inclusion complexes.

Figure 2. Distinctive appearance between amylose and amylose-PTHF complexes after 16 h rotation at 85 °C followed by sedimentation at 85 °C for 1 h (top) and 2 h (bottom).

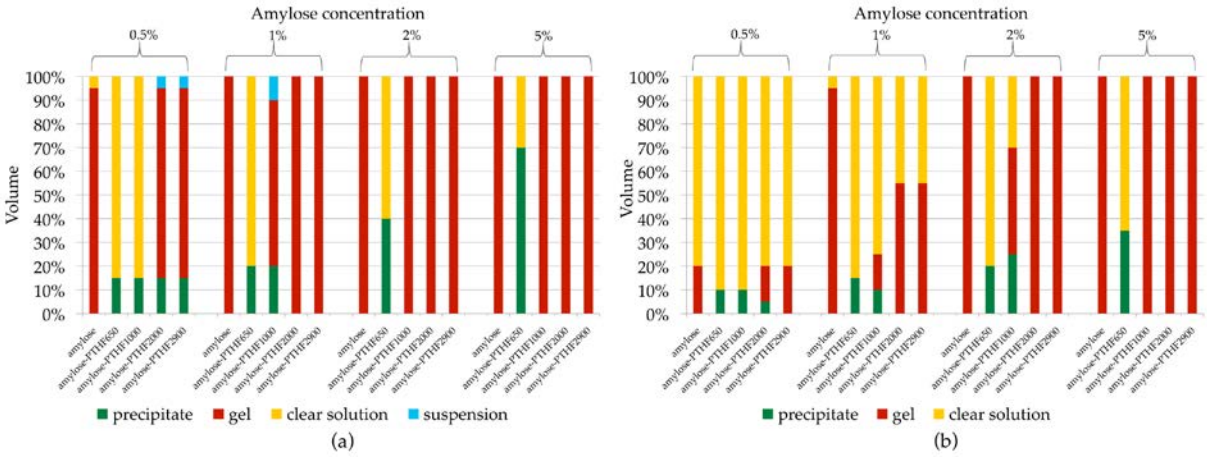

Figure 3. Estimation on gelation and sedimentation behavior of amylose-PTHF complexes after more than 16 h at room temperature (a) and after additional centrifugation for 30 min at 1000 rpm (b).

Table 1. Estimation of uncomplexed amylose for amylose-PTHF complexes.

Guest molecules	Reference (complex)[a]	Reference (amylose)[a]	Gel volume (complex)	Uncomplexed amylose	Uncomplexed amylose
PTHF650	Figure 3(a) (5%) no gel volume	Figure 3(a) (0.5%) gel 95%	0 < 95%	< 10%	< 5.3%
	Figure 2 (5%) precipitate 50%	Figure 3(a) (0.5%) gel 95%	[50/95]x[0.5/5]x 100%	< 5.3%	
PTHF1000	Figure 3(a) (1%) gel volume 70%	Figure 3(a) (0.5%) gel 95%	70% < 95%	< 50%	25-50%
	Figure 3(b) (2%) gel volume 45%	Figure 3(b) (0.5%) gel volume 20% and Figure 3(b) (1%) gel volume 95%	20% < 45% < 95%	25-50%	
PTHF2000 PTHF2900	Figure 3(b) (1%) gel volume 55%	Figure 3(b) (0.5%) gel volume 20%	55% > 20%	> 50%	> 50%

[a]Figure 2 and 3 from the Supporting Information section are used as references.

Table 2. DSC data of inclusion complexes between amylose and PTHF.

Sample	Run (°C)	Heating scan			Cooling scan		
		Onset (°C)	Peak (°C)	Δ*H* (J/g)	Onset (°C)	Peak (°C)	Δ*H* (J/g)
Method A							
PTHF1100[a]	1 (120)	21.4	23.3	21.8	10.9	7.7	-19.4
	2 (140)	20.9	23.5	23.4	10.6	7.7	-19.9
	3 (160)	20.1	23.1	22.9	10.3	7.5	-18.6
Potato amylose with							
5% PTHF1100	2 (140)	120.3	128.2	6.9	106.5	101.4	-5.0
	3 (160)	130.0	138.3	5.3	104.4	100.4	-1.6
20% PTHF1100	2 (140)	117.5	124.8	18.2	111.7	105.9	-17.5
	3 (160)	125.6	133.8	21.1	105.5	101.1	-12.8
20% PTHF650	3 (160)	126.0	132.3	17.7	105.0	97.8	-22.3
20% PTHF1000	3 (160)	128.4	133.6	14.2	106.6	100.5	-13.6
20% PTHF2000	3 (160)	130.4	134.6	2.7	108.4	102.2	-3.3
Method B							
Potato amylose with							
20% PTHF650	3 (160)	126.0	133.3	19.9	104.0	95.7	-24.8
20% PTHF1000	3 (160)	126.9	134.1	15.0	106.7	100.5	-16.0
20% PTHF2000	3 (160)	129.5	134.8	4.5	105.1	97.8	-4.8
Synthetic amylose with							
20% PTHF650	3 (160)	122.7	130.8	9.5	107.0	101.2	-14.6
20% PTHF1000	3 (160)	123.8	130.8	8.8	104.6	99.1	-7.6
20% PTHF2000	3 (160)	123.5	130.6	2.9	102.1	97.4	-2.6
20% PTHF1100	3 (160)	124.3	135.2	15.7	109.5	104.3	-9.2
20% PTHF650	4 (160)	127.1	133.4	19.3			
20% PTHF1000	4 (160)	125.3	134.5	14.2			
20% PTHF2000	4 (160)	119.4	134.0	10.0			
20% PTHF1100	4 (160)	122.9	135.5	18.0			
Method C[b]	1 (160)				60	40	(19-40)
Potato amylose	1 (160)						
Potato amylose with							
20% PTHF650	1 (160)	129.0	134.2	26.8	105.5	98.6	-17.2
20% PTHF1000	1 (160)	129.7	136.9	10.5	98.3	92.6	-8.7
20% PTHF2000	1 (160)				92.6	85.9	-3.7
20% PTHF2900	1 (160)				93.3	88.1	-1.5

[a]Measured as 2%(w/w) concentration in water. [b]Measured as 5% (w/w) concentration in water. The rest of the DSC data were obtained as 10% (w/w) concentration in water. The amount of the guest PTHF was calculated based on amylose (w/w).

Table 3. DSC data of first heating scan of inclusion complexes between synthetic amylose and PTHF (method B).

Day	Amylose-PTHF650			Amylose-PTHF1100		
	Onset (°C)	Peak (°C)	ΔH (J/g)	Onset (°C)	Peak (°C)	ΔH (J/g)
0	123.8	131.9	20.2	124.1	136.6	18.7
1	125.4	131.4	24.0	127.3	137.4	22.9
7	126.0	131.5	24.2	128.1	137.3	21.7

Measured for 20% (w/w) PTHF concentration based on amylose. The measurements were conducted to study the stability of amylose-PTHF complexes.

Table 4. XRD data of amylose-PTHF complexes.

Sample	Diffraction peaks							
	2θ (°)/ *d* (nm)	Plane (*hkl*)	2θ (°)/ *d* (nm)	Plane (*hkl*)	2θ (°)/ *d* (nm)	Plane (*hkl*)	2θ (°)/ *d* (nm)	Plane (*hkl*)
Amylose[a]	17.1/0.52		21.7/0.41					
PTHF	19.9/0.45		24.4/0.36					
Amylose inclusion complexes with								
PTHF650	13.1/0.68	200	(18.4)/0.48	221	21.4/0.42	450*	23.7/0.38	550*
	17.3/0.51	A	19.8/0.45	310	22.4/0.40	002^{A}	(25.0)/0.36	340
PTHF-b650	17.2/0.52	A	22.2/0.40	A	(25.9)/0.34	422*		
	19.6/0.45	P	23.9/0.37	222*	(28.6)/0.31	411		
PTHF1000	13.4/0.66	410*	20.0/0.44	310	23.7/0.38	222*	(28.5)/0.31	411
	17.3/0.51	A	21.4/0.42	450*	24.4/0.36	P		
	18.7/0.47	221	22.5/0.40	002^{A}	(27.3)/0.33	222		
PTHF2000	13.3/0.67	111	(18.4)/0.48	221	(21.4)/0.42	450*	24.3/0.37	P
	17.2/0.52	A	19.9/0.45	310^{P}	22.2/0.40	A	28.6/0.31	411
PTHF2900	17.3/0.51	A	20.0/0.44	310^{P}	22.5/0.40	A	24.4/0.36	P
	(18.4)/0.48	221	(21.4)/0.42	450*	(23.8)/0.37	222*		

[a]The amylose was solubilized and freeze-dried. A and P denote amylose and PTHF. **hkl* values of the diffracting planes are determined based on the orthorhombic unit cell of an amylose-*n*-butanol/*n*-pentanol complexes,[21] while the rest are calculated based on amylose-fatty acid complexes.[15] The *d*-spacing values are calculated based on Bragg's law for n = 1. The data in brackets are for shoulder-shaped peaks.