Yttrium Sulfate Recovery Using Ethanol as Antisolvent in a Fluidized Bed Reactor

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2.5. Initial trial experiments

Fluidization equations:

The Ergun equation in Eq.1 used to calculate the minimum fluidisation velocity (u_{mf}):

$$\frac{\rho(\rho_s - \rho)gd^3}{\mu^2} = 150\left(\frac{1-e_{mf}}{e_{mf}^3}\right)\left(\frac{u_{mf}d\rho}{\mu}\right) + \left(\frac{1.75}{e_{mf}^3}\right)\left(\frac{u_{mf}d\rho}{\mu}\right)^2 \quad (1)$$

which can further be refined with the Galileo number (Ga) and Reynolds number (Re'_{mf}):

$$\frac{\rho(\rho_s - \rho)gd^3}{\mu^2} = Ga \quad (2)$$

$$\frac{u_{mf}d\rho}{\mu} = Re'_{mf} \quad (3)$$

$$Ga = 150\left(\frac{1-e_{mf}}{e_{mf}^3}\right)Re'_{mf} + \left(\frac{1.75}{e_{mf}^3}\right)Re'^{\,2}_{mf} \quad (4)$$

where g is the gravitational acceleration, with ρ_s *and* ρ as the particle- and fluid- density respectively for a bed, e_{mf} is the bed voidage estimated to be 0.4, d is the particle diameters as meters, and μ is the liquid viscosity as cP.

The maximum fluidization velocity can also be calculated for the point at which the particles will be elutriated:

$$u_{max} = \frac{d^2 g}{18\mu}(\rho_s - \rho) \quad (5)$$

Reynolds number (*Re)* equation:

$$Re = \frac{\rho VD}{\mu} \quad (6)$$

Where ρ is the fluid density (kg/m³), V is the superficial velocity (m.s^{-1}), D is the inlet diameter (m) and μ is the dynamic viscosity (Pa·s).

Table S1: Reynolds number at all inlet ports

	Total Inlet Flow Rates* (ml/min)	O/A ratio	Aq. Y^{3+} (ml/min)	Ethanol (ml/min)	Water (Re)	Ethanol (Re)	Mixture (Re)
Trial	80	0.9	37.89	42.11	160	118	197
	20	*1.1	9.47	10.53	40	29	98
	40	*1.0	18.95	21.05	80	59	197
	80	0.9	37.89	42.11	160	118	989
	200	0.9	94.74	105.26	401	296	1004
	200	1.0	100.00	100.00	423	281	1021
	200	1.1	104.76	95.24	443	267	1007
	200	0.56	71.79	128.21	303	360	395

* O/A correction

Table S2: The crystal sizes elutriated and recirculated.

Recirculation rate (mL/min)	O/A ratio	Minimum elutriation size (μm)
20	1.1	27
40	1.0	39
80	0.9	54
170	0.9 – 1.1	79

3.2. Initial trial experiments

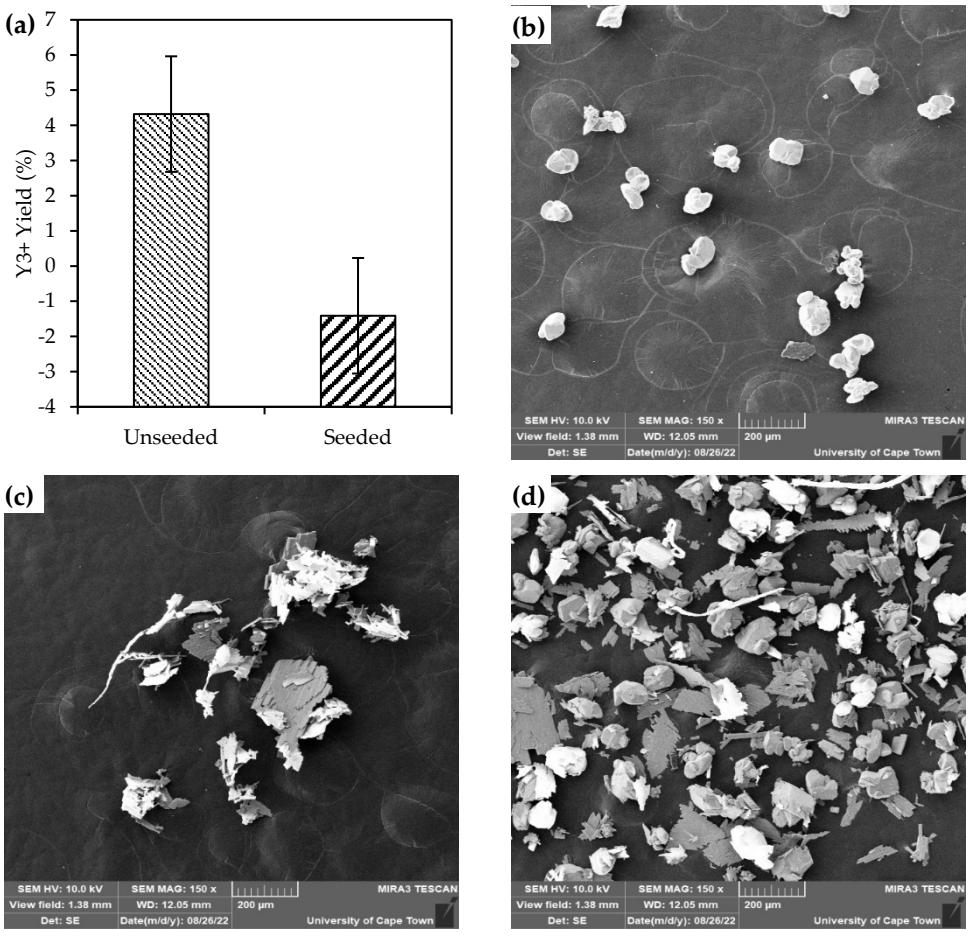


Figure S1. (a) The recovery of yttrium in a continuous FBR after a period of 30 min at O/A = 0.9 at a total flow rate of 80 ml/min, (b) $Y_2(SO_4)_3.8H_2O$ reagent seeds, (c) crystallized product without seeding, and (d) the seeded crystallized product.

3.3.1. The effect of equated flow rates

Table S3: The effect on flow rate changes from the bottom to the top of the FBR

Total Inlet Feed Rates (ml/min)	Eth (ml/min)		Aq. Y3+ (ml/min)		O/A ratio	
	Bottom	Top	Bottom	Top	Bottom	Top
20	9.47	10.43	10.53	9.57	0.9	1.1
40	18.95	19.63	21.05	20.37	0.9	1.0
80	37.89	38.52	42.11	41.48	0.9	0.9

Table S4. Adjusted experimental variables

	$[Y^{3+}]$ (g/L)		**O/A**			**Feed Rate (mL/min)**				**Recirculation (mL/min)**			
Exp	**0.4**	**0.87**	**0.56**	**0.9**	**1.0**	**1.1**	**20**	**40**	**80**	**200**	**20**	**40**	**80**	**170**
A.1		☑		☑ →		*	☑				☑			
A.2		☑		☑ →	*			☑				☑		
A.3		☑		☑					☑				☑	
B.1		☑		☑ →		*	☑					☑		
B.2		☑		☑ →		*	☑						☑	
C.1		☑		☑ →	*			☑			☑			
C.2		☑		☑					☑		☑			
D.1		☑	☑							☑				☑
D.2		☑		☑						☑				☑
D.3		☑			☑					☑				☑
D.4		☑				☑				☑				☑
E.1	☑		☑							☑				☑
E.2	☑			☑						☑				☑

* Total effective amount as further explained in Section 3.3.

3.3.1. The effect of equated flow rates

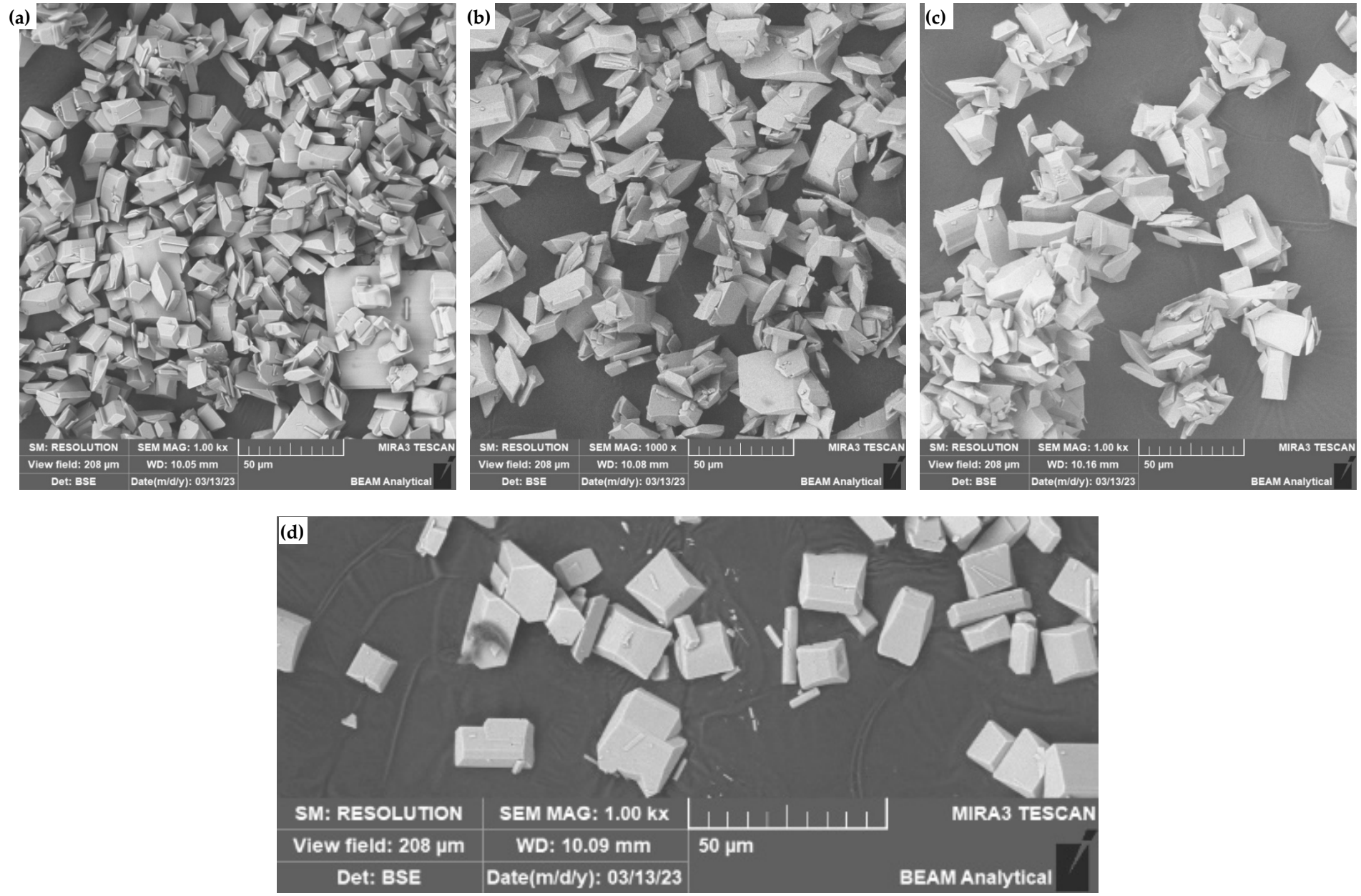


Figure S2. SEM images at the equated flow rates of (a) 20 mL/min, (b) 40 mL/min and (c) 80 mL/min. Existence of fines (d).

3.3.2. The effect of total inlet flow rates

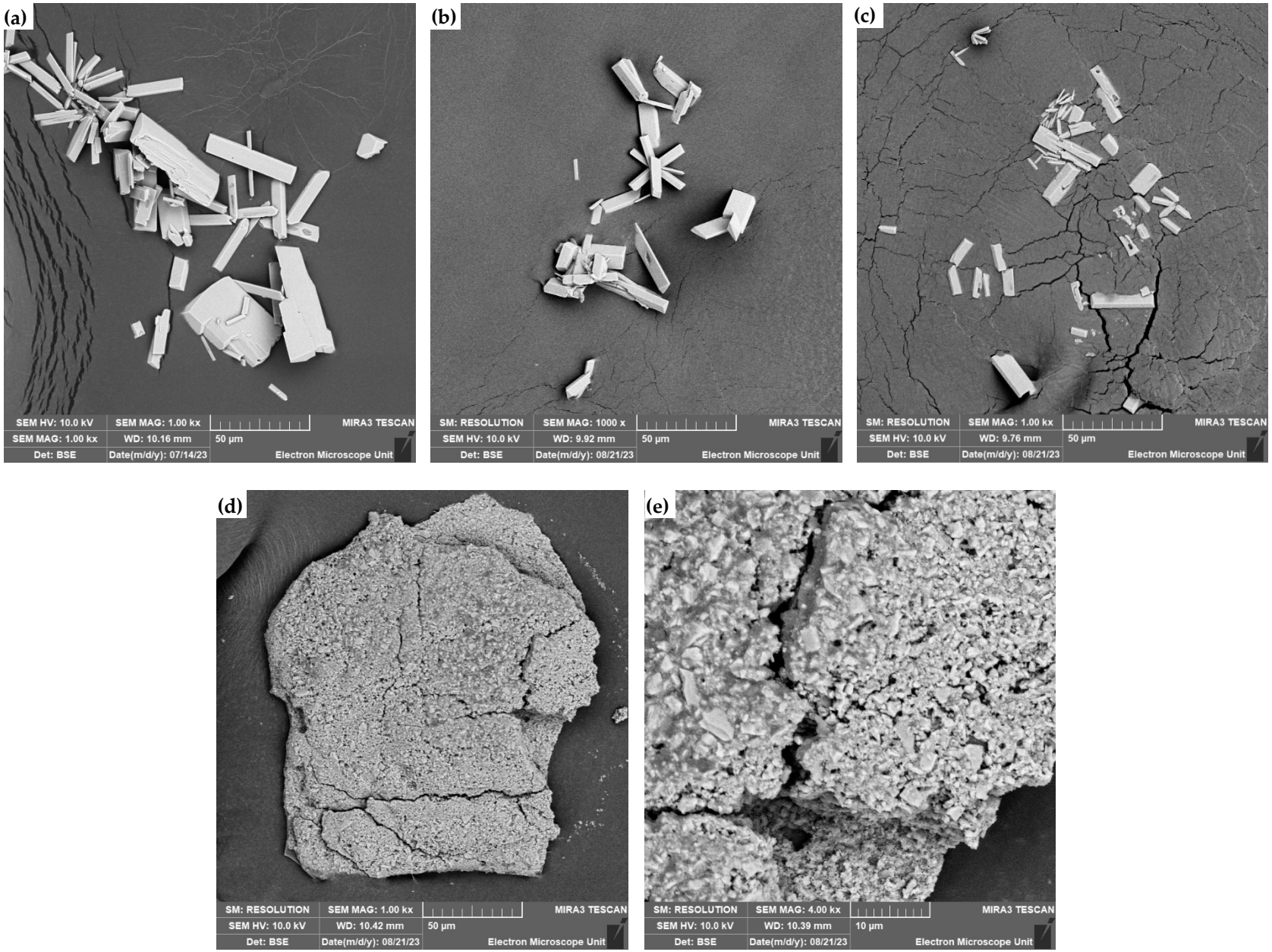


Figure S3. SEM images with the total inlet flow rates for the final product at (a) 20 mL/min, (b) 40 mL/min and (c-e) 80 mL/min after the inlet period.

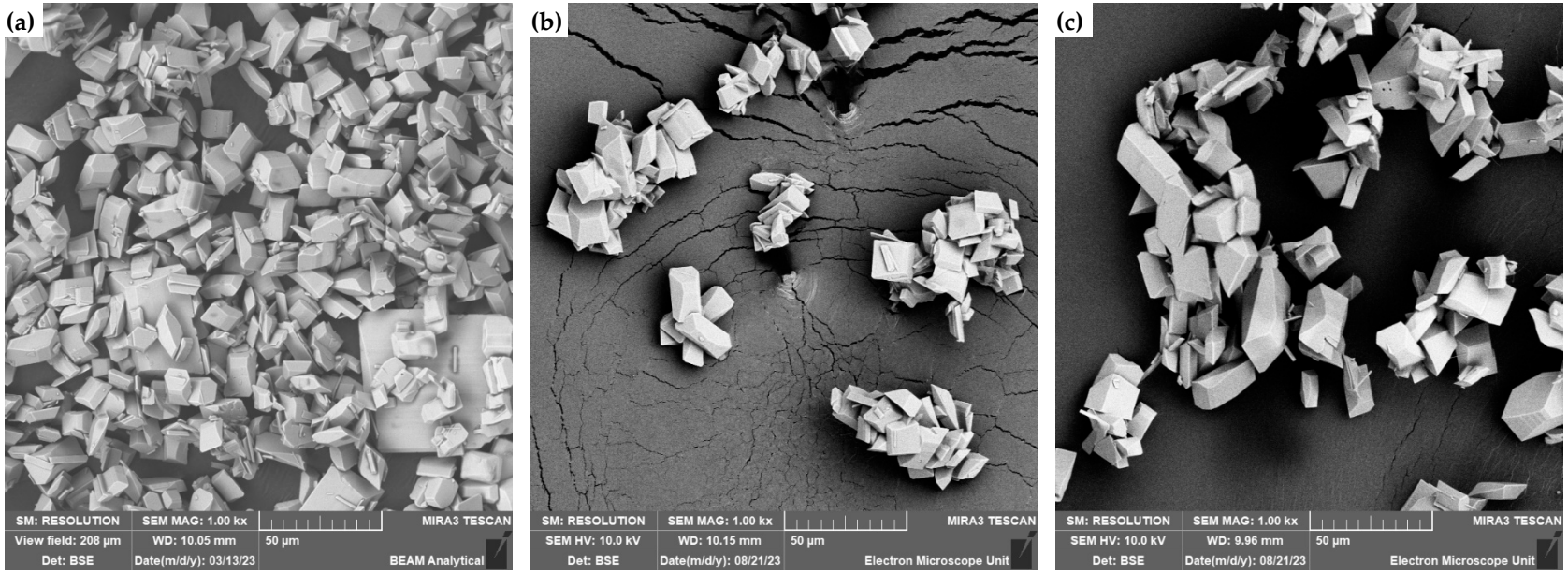


Figure S4. SEM images with the total inlet flow rates for the final product at (a) 20 mL/min, (b) 40 mL/min and (c) 80 mL/min for the final product.

3.3.3. The effect of recirculation rate

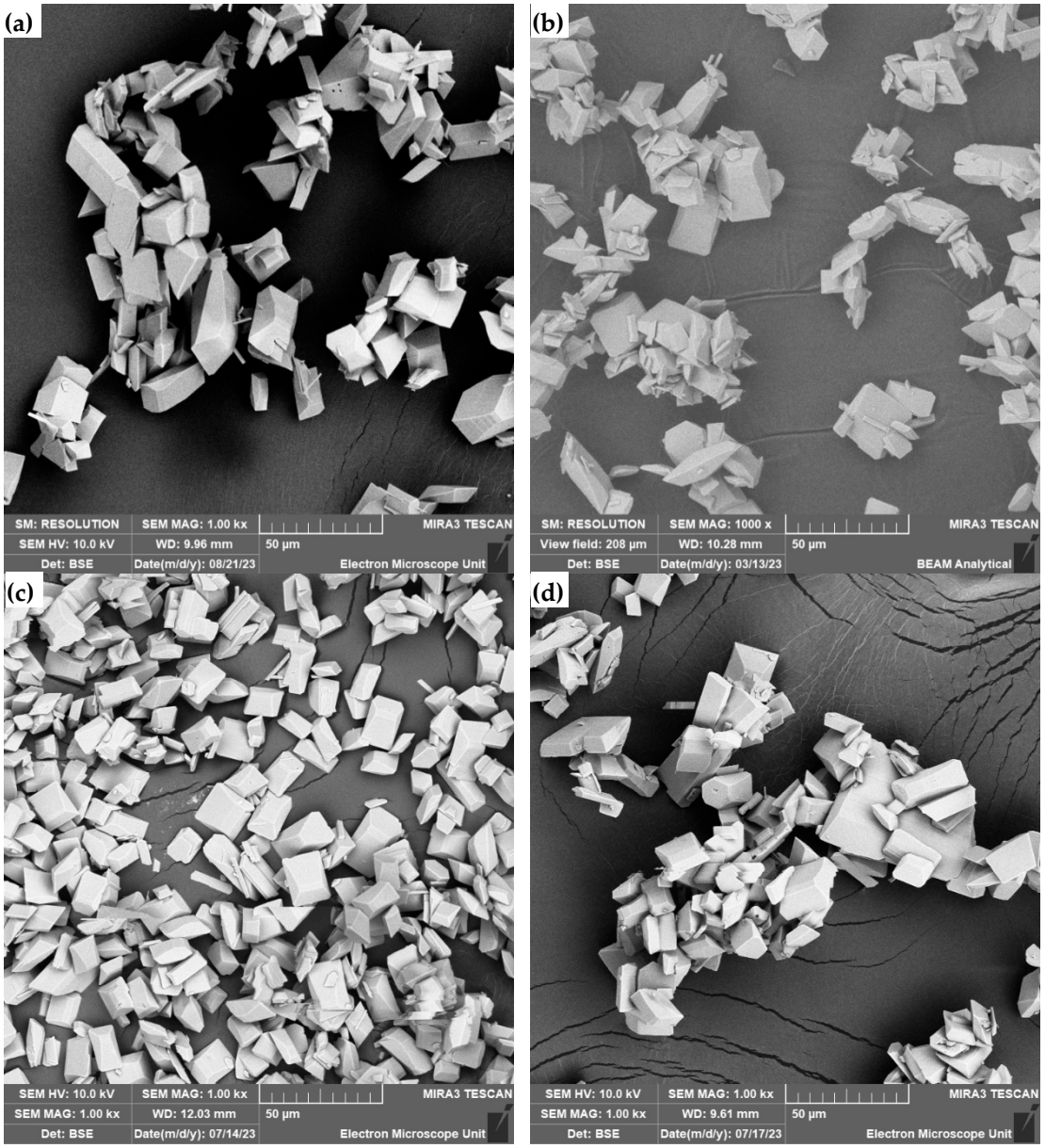


Figure S5. SEM images with the recirculation rate for low S at (a) 20 mL/min and (b) 80 mL/min, and high S at (c) 20 mL/min and (d) 80 mL/min.

3.3.4. The effect of O/A ratio

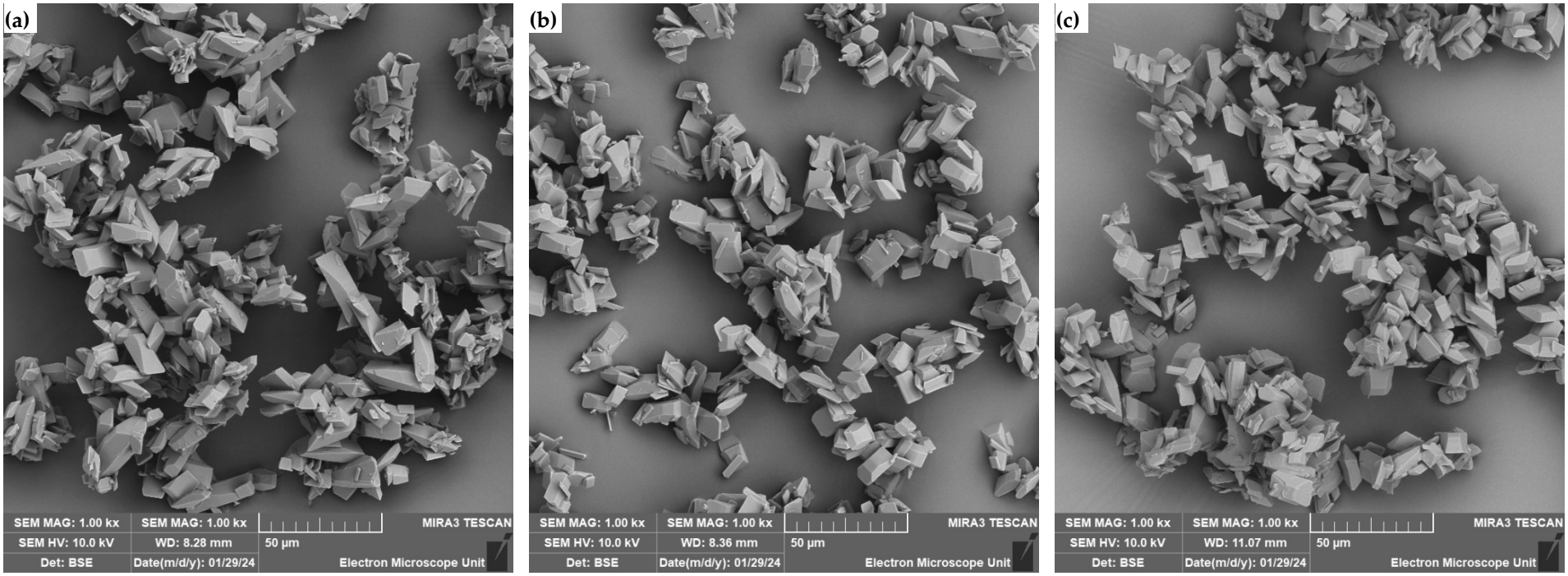


Figure S6. SEM images with the O/A ratios at (a) 0.9, (b) 1.0 and (c) 1.1.

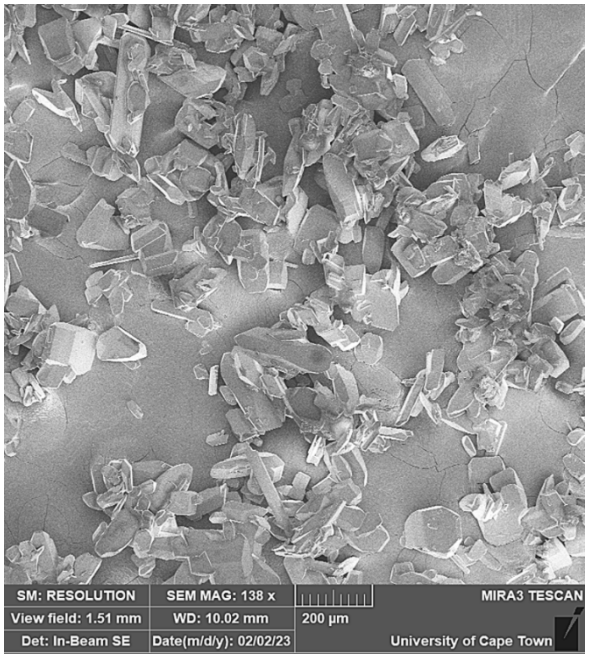


Figure S8. SEM image of the crystalline product formed during the storage period.

Additional stirred reactor experiments:

Method:

Ethanol was added at the respective O/A ratios of 0.9, 1.0, 1.1 and 1.2 to 10 mL of the aqueous Y^{3+} solution as a single immediate batch addition. The mixture was stirred at 250 rpm using a magnetic bar for 15 min followed by immediate filtration. An additional experiment was done at O/A = 0.9 and stirred for 24h. The further sample treatment was done the same as in the main text.

Results:

In **Figure S7**, it was seen that at the end of 15 min reaction time an amorphous product existed at 0.9 and 1.0 with yields of 4% and 8%, respectively. Whereas an O/A ratio = 1.1 resulted in a crystalline product with yield at 56%. Although for O/A = 0.9, large crystals were observed after stirring 24h with a yield of 87%.

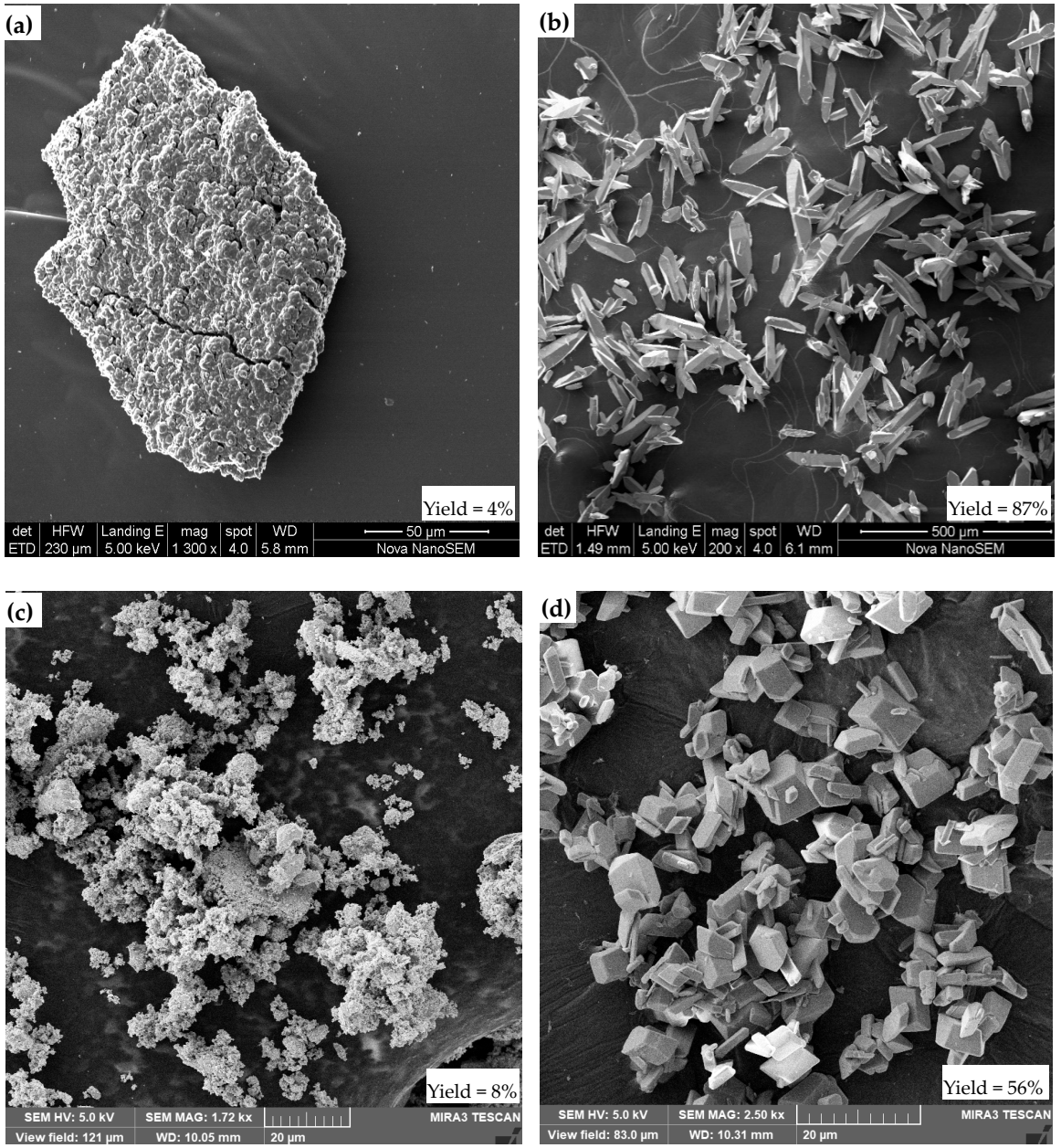


Figure S8. SEM images of the stirred batch reactor experiments with (a-b) O/A = 0.9 respectively at 15 min and 24, (c) O/A = 1.0 and (d) O/A = 1.1.