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Separation and characterization of clays and ultrafines from commercial streams of a naphthenic froth treatment process

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OUTLINE FOR THE TALK

- Previous Work: Quantitative Separation and Characterization of Solids in NFT Streams Based on Solids Wettability
- Present Work: Quantitative Separation and Characterization of <2μm Clay and <0.2μm Ultrafine Solids in NFT Streams
- Samples Studied: Seven NFT Streams from Commercial Plant
- Quantitative Separation Technique for Clays and Ultrafines
- Evolution of Clay and Ultrafine Contents for the NFT Streams
- Elemental / Mineralogical Compositions of Clays and Ultrafines
- Conclusions

Previous Work: Quantitative Separation and Characterization of Solids in NFT Streams Based on Solids Wettability

many replicates

sample (~10g)
+
toluene (~70g)
+
distilled water
(~15g)

1. Agitate then centrifuge,
2. Collect supernatant
3. Add fresh toluene aliquots
4. Repeat step 1&2
5. Transfer T/W interfacial solids to a next jar
6. Repeat steps 1-4 on jar in step 5; etc.

Solids from NFT streams (froth, products) isolated into 4 wettability fractions :

- Hydrophilic Solids (HPS)
- Intermediate Solids (IS)
- Organic Rich Solids (ORS)
- High Speed Centrifugation Solids (HSCS)

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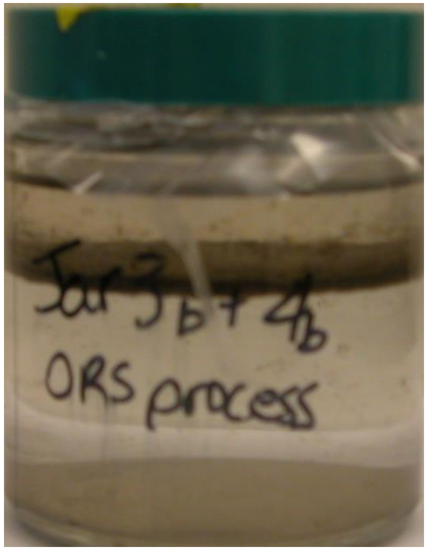


Solids in **froths**: ~50 wt% ORS, ~35 wt% HPS, ~5–10 wt % IS, <5 wt% HSCS
HPS: ~40–55 wt% QTZ+SIL, ~30–45 wt% CLAY, ~5 wt% HEAVY, <5 wt% ORC
ORS: ~10–15 wt% QTZ+SIL, ~15–25 wt% CLAY, ~40 wt% HEAVY, ~15–20 wt% ORC

Solids in **products**: ~60 wt% ORS, ~35 wt% HPS, ~5 wt % IS, <5 wt% HSCS
HPS: ~10–15 wt% QTZ+SIL, ~40–60 wt% CLAY, ~10–15 wt% HEAVY, <5 wt% ORC
ORS: <5 wt% QTZ+SIL, ~35–40 wt% CLAY, ~25–35 wt% HEAVY, ~10–15 wt% ORC

OBJECTIVE FOR PRESENT WORK

For the first time, quantify the absolute amounts and compositional properties of <2μm clay and <0.2μm ultrafine solids in industrial streams from the NFT process used at commercial mined oil sands operations

Samples Studied

Representative samples were collected and analyzed from process streams at commercial NFT plant.

The sample suite comprised:

- ❖ One bitumen froth (F1)
- ❖ Two solvent-diluted bitumen products (P1, P2)
- ❖ Four tailings (T1, T2, T3, T4)

Particle size fractions commonly employed in the mineralogical, chemical and physical analysis of soils (after Jackson 1969)

Size fraction name	Particle size range	Technique of obtaining fraction
very coarse sand	1000−2000 µm	Sieve round-hole, on 1-mm, through 2-mm
coarse sand	500−1000 µm	Sieve, round-hole, on 0.5-mm
medium sand	250−500 µm	Sieve, screen, on 0.25-mm (60 meshes per inch)
fine sand	100−250 µm	Sieve screen, on 0.1-mm (140 meshes per inch)
very fine sand	50−100 µm	Sieve, screen, on 0.05-mm (300 meshes per inch)
coarse silt	20−50 µm	Sieve, decantation
medium silt	5−20 µm	Decantation, centrifuge
fine silt	2−5 µm	Decantation, centrifuge
coarse clay	0.2−2 µm	Decantation, centrifuge
medium clay	0.08−0.2 µm	Decantation, centrifuge
fine clay	<0.08 µm	Decantation, (super)centrifuge

Separation of clays and ultrafines: application of Stoke's law to isolate <2 and <0.2 μm solids

Jackson's Book, Eq. 3.7, p.127:

$$t_{min} = \frac{63.0 * 10^8 * n * log_{10}\frac{R}{S}}{N_m^2 * \ D_u^2 * \Delta s}$$

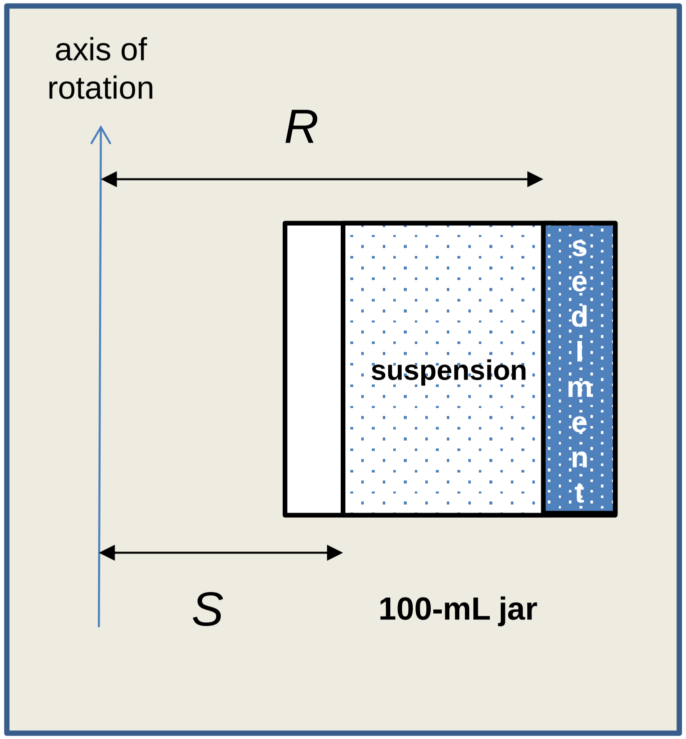
$$N_m^2 = rpm\ centrifuge$$

$$D_u^2 = particle\ diameter\ in\ microns$$

$\Delta s = difference\ in\ specific\ gravity\ between\ solvated\ particle\ and\ the\ suspension\ liquid$ (in g/cm³)

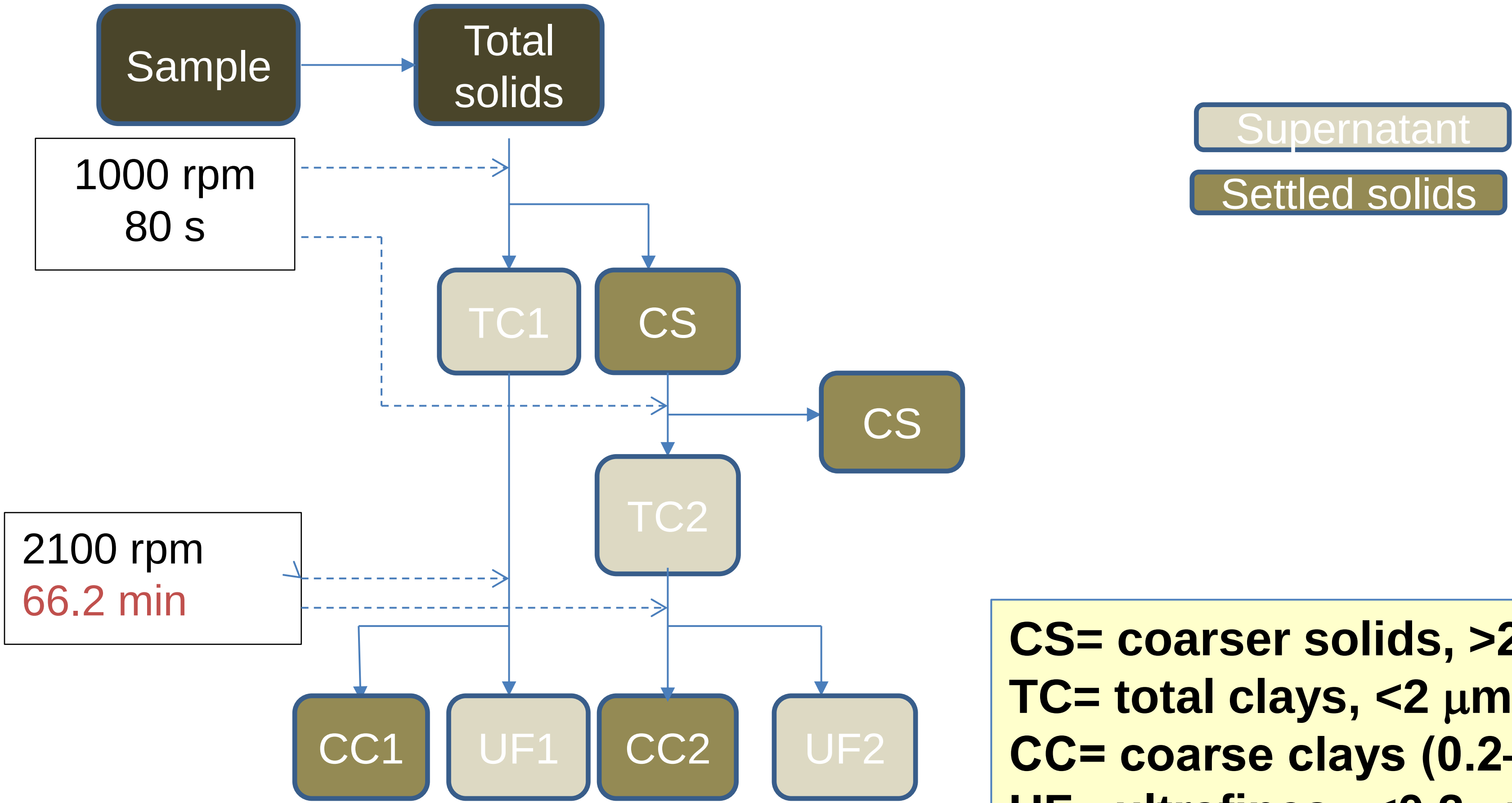
$\frac{R}{S} = ratio\ of\ distance\ from\ top\ of\ sediment\ to\ centre\ of\ centrifuge\ (R)\ to\ that\ of\ top\ of\ suspension\ to\ centre\ of\ centrifuge\ (S)$

$n = viscosity\ of\ liquid\ in\ poises\ based\ on\ temperature$ (in poise)



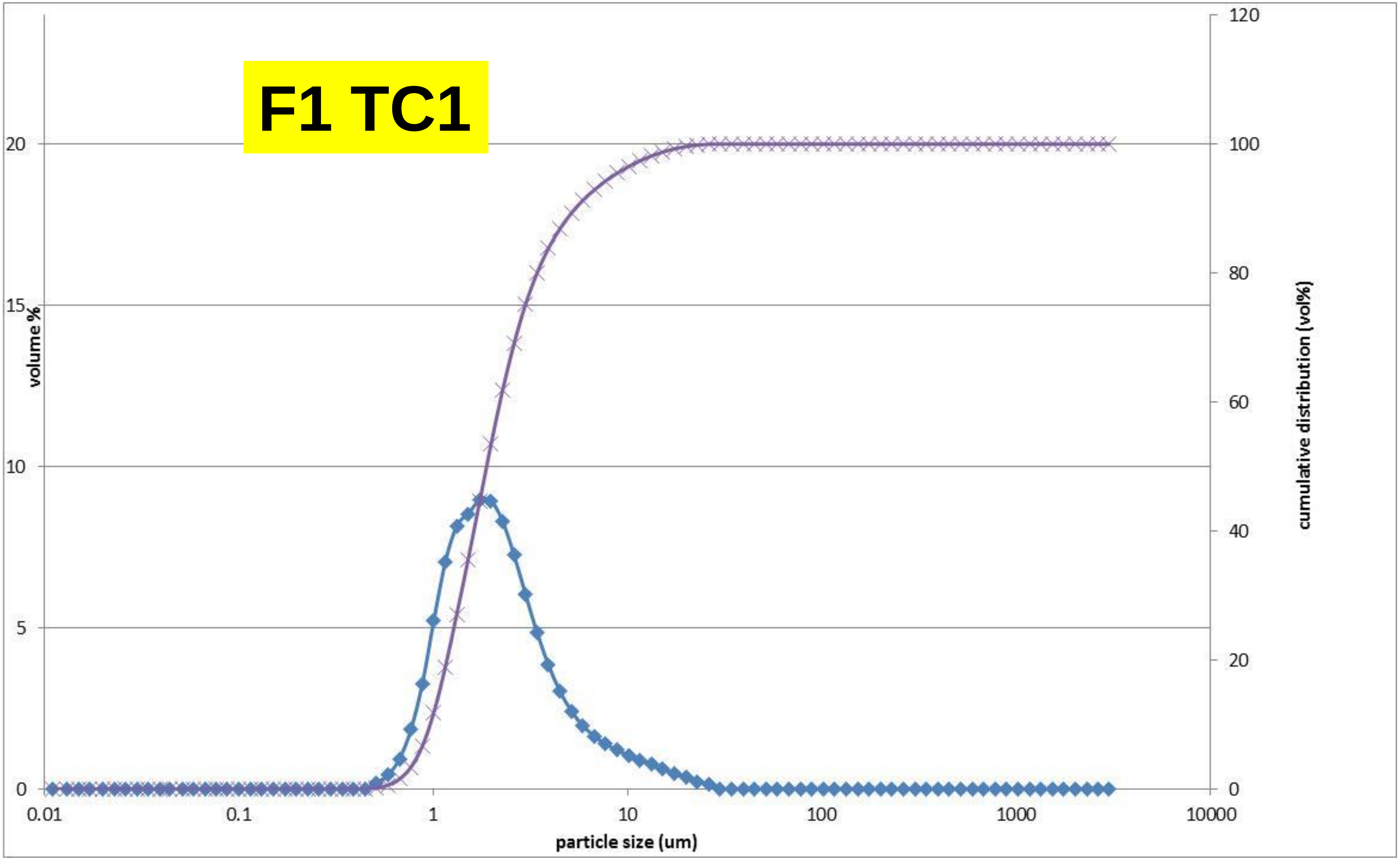
For siliceous rock like oil sands, a particle density ρ_p of 2.65 kg/m³ is adequate for all diameters down to 0.2 μm. A specific gravity of 2.5 kg/m³ is appropriate for siliceous particles in the size range of 0.2 μm. In the <0.2 μm fraction the specific gravity in heavy liquids falls to about 2.2 kg/m³. Water has a density of 0.998 kg/m³ at 20 °C.

Analytical procedures optimized for quantitative clay-ultrafines separation technique



CS= coarser solids, >2 µm
TC= total clays, <2 µm
CC= coarse clays (0.2–2 µm)
UF= ultrafines, <0.2 µm

Laser scattering PSD analyses of solids suspensions of separated clays and ultrafines



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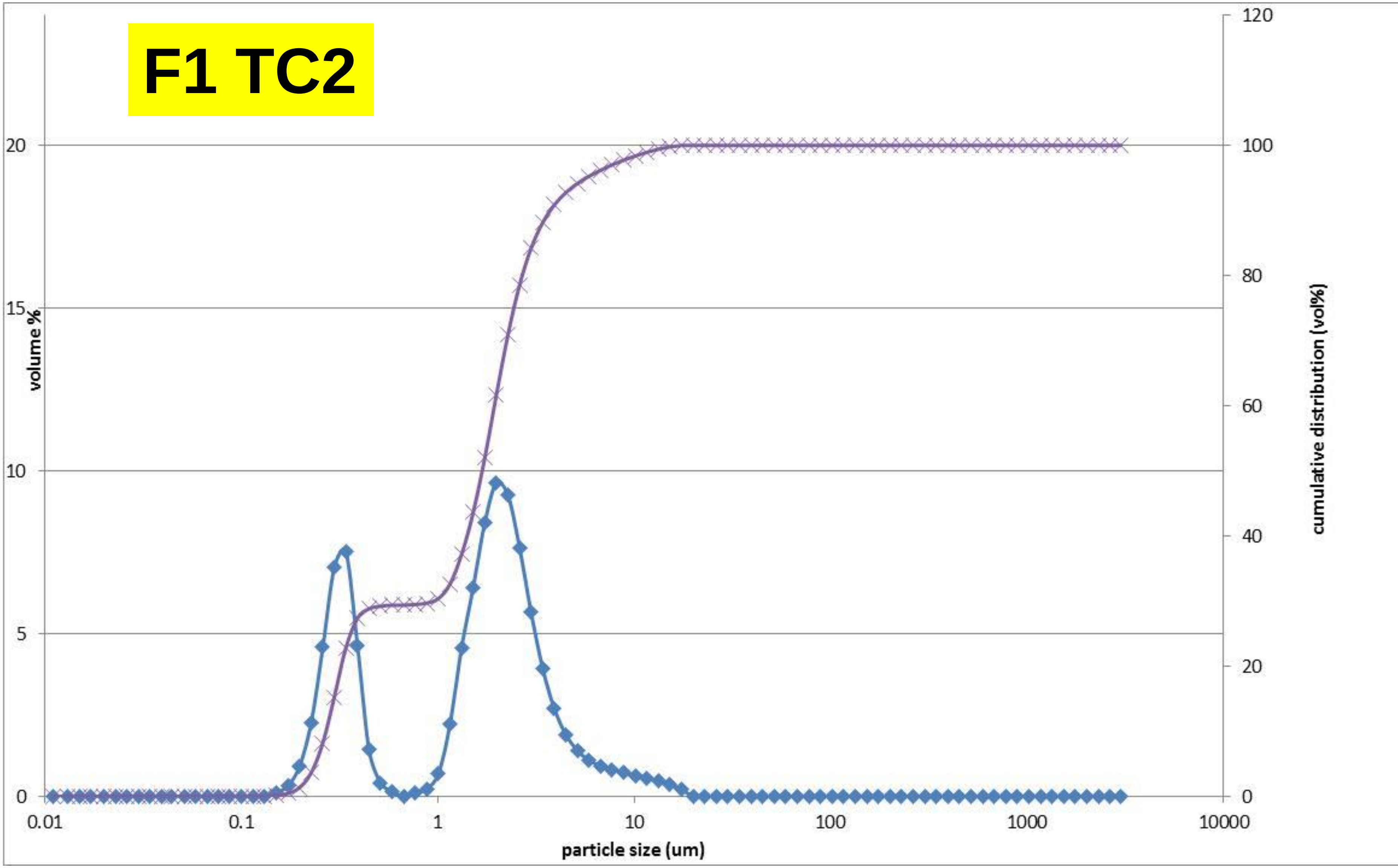
Laser scattering PSD analyses of solids suspensions of separated clays and ultrafines



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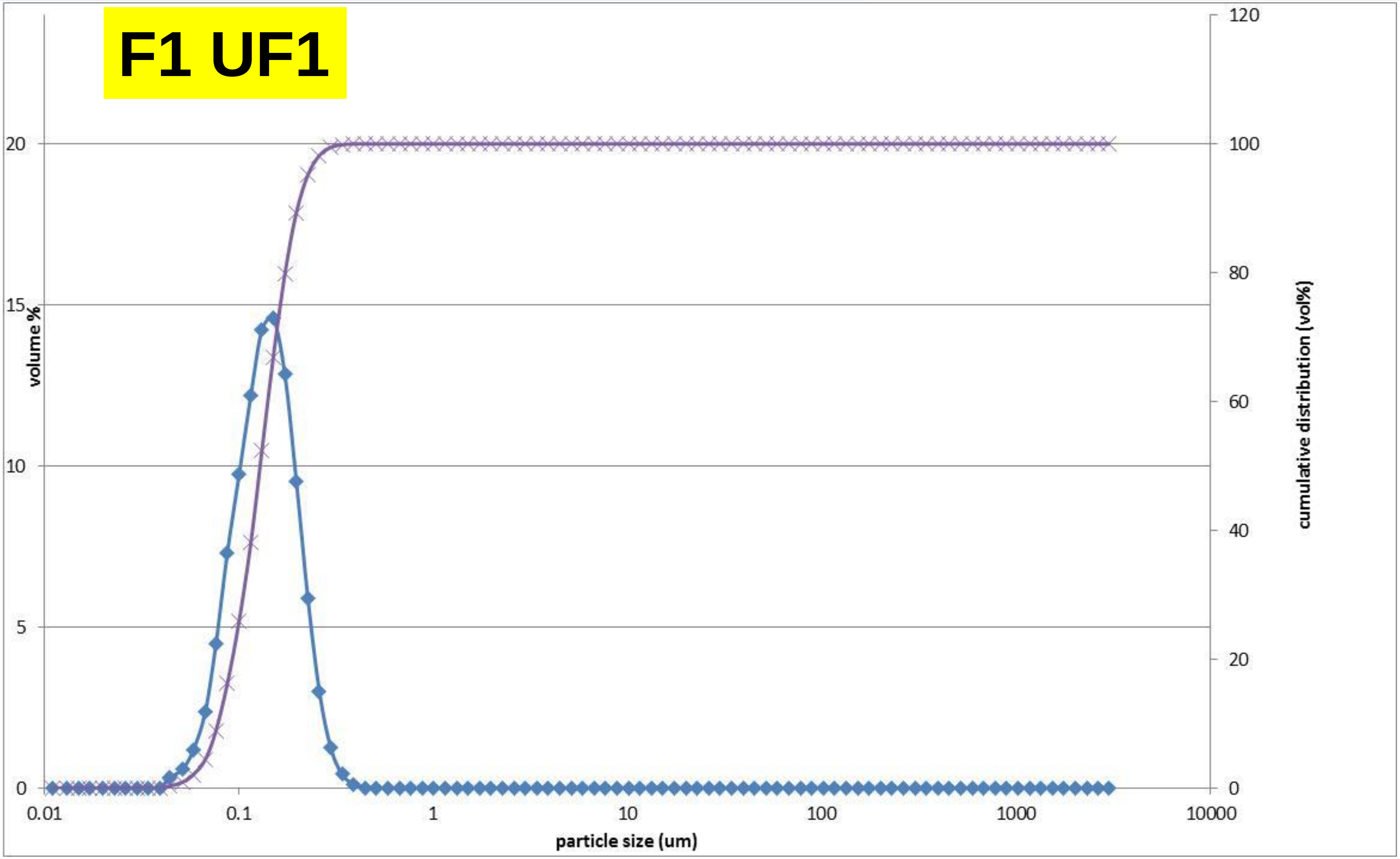
Laser scattering PSD analyses of solids suspensions of separated clays and ultrafines



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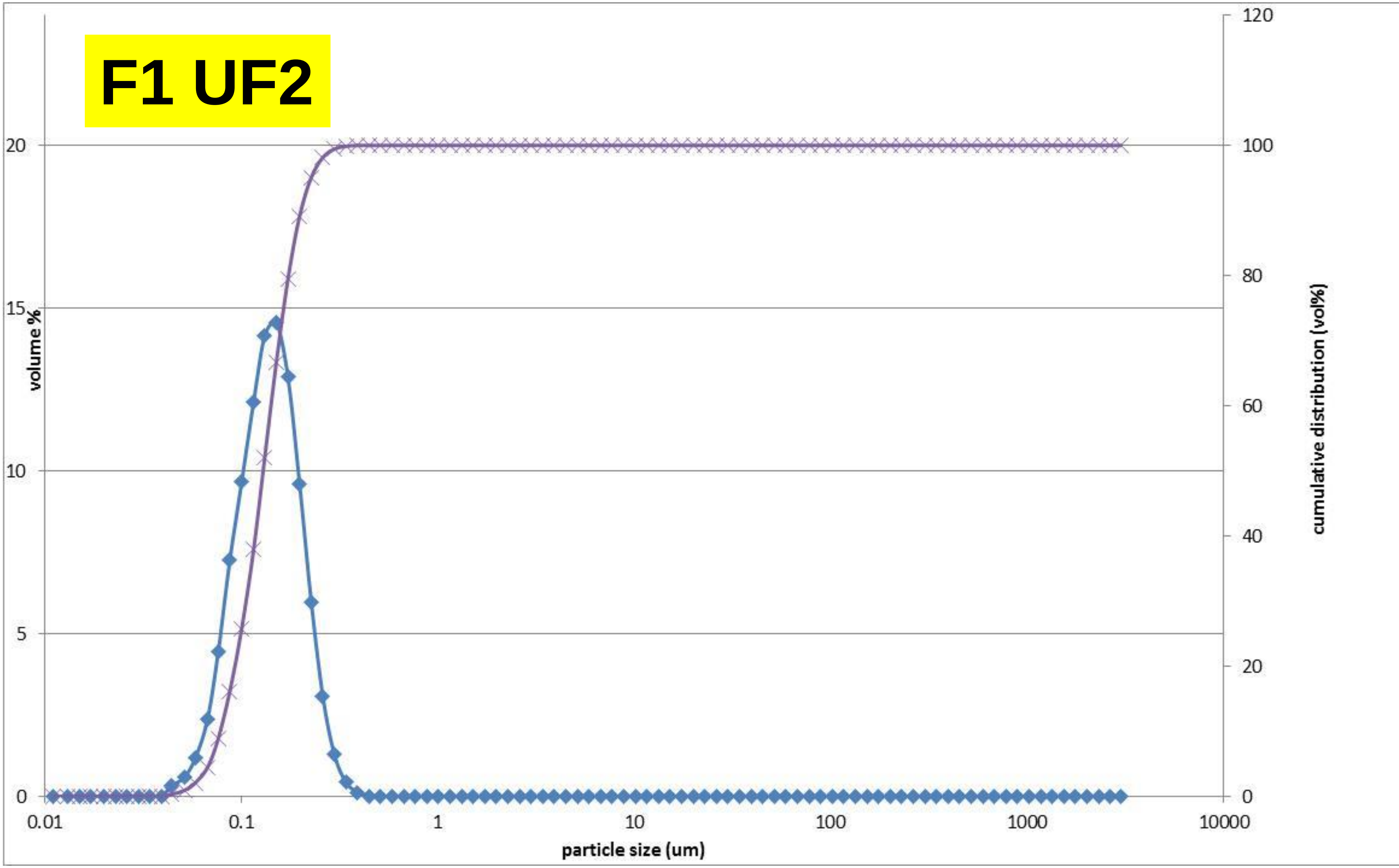
Laser scattering PSD analyses of solids suspensions of separated clays and ultrafines



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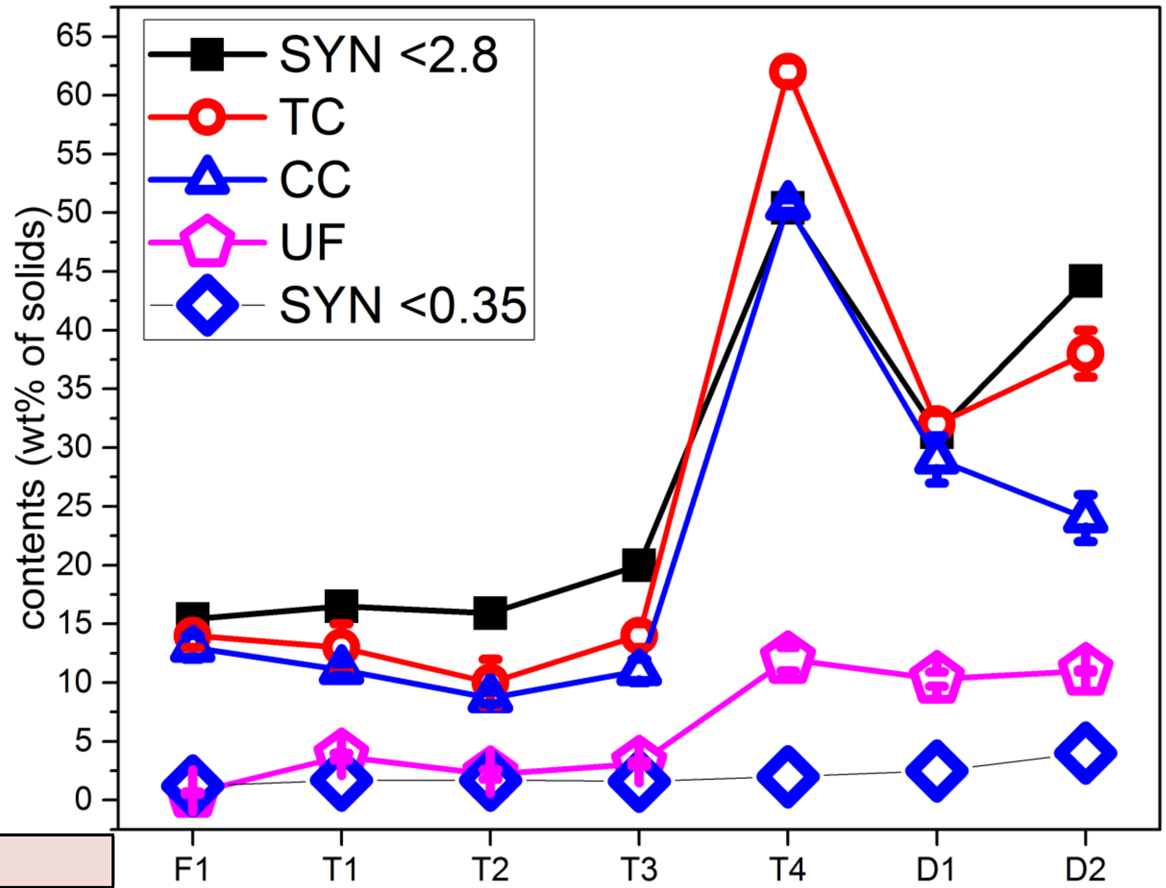
Evolution of Total Clays, Coarse Clays, and Ultrafines Contents

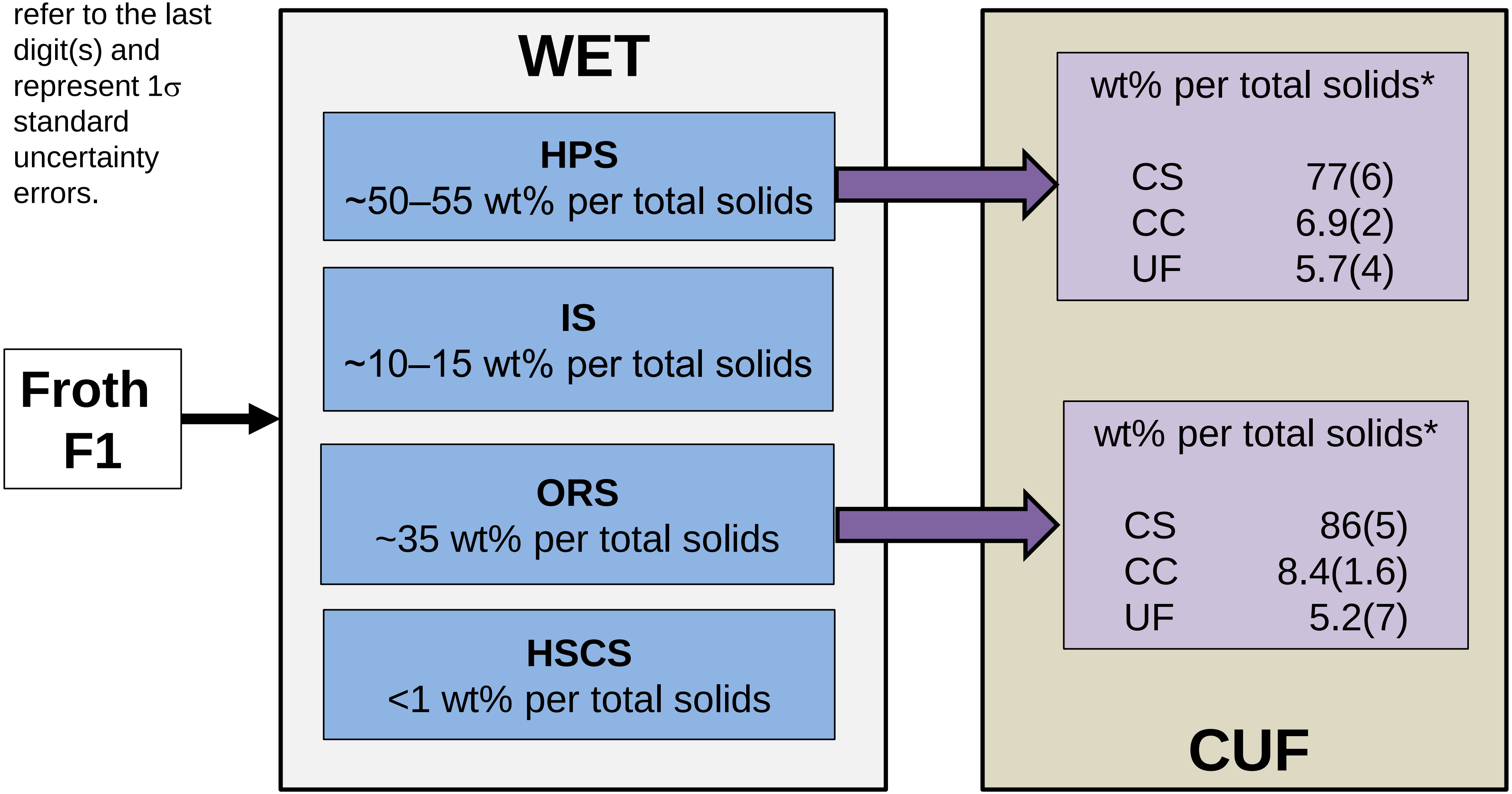


	wt% per total solids*					
	SYN <2.8	TC	CS	CC	UF	SYN <0.35
F1	15.4	14(1)	84(2)	13(1)	0.6(1)	1.2
T1	16.5	13(2)	85(3)	11.1(5)	3.7(3)	1.7
T2	15.9	10(2)	89(3)	8.7(5)	2.2(4)	1.7
T3	20.0	14(1)	85(2)	11(1)	3.1(2)	1.6
T4	50.4	62(1)	37.1(4)	50.6(5)	12(1)	2.0
D1	31.3	32(1)	59(3)	29(2)	10.3(6)	2.5
D2	44.2	38(2)	55(4)	24(2)	11(1)	4.0

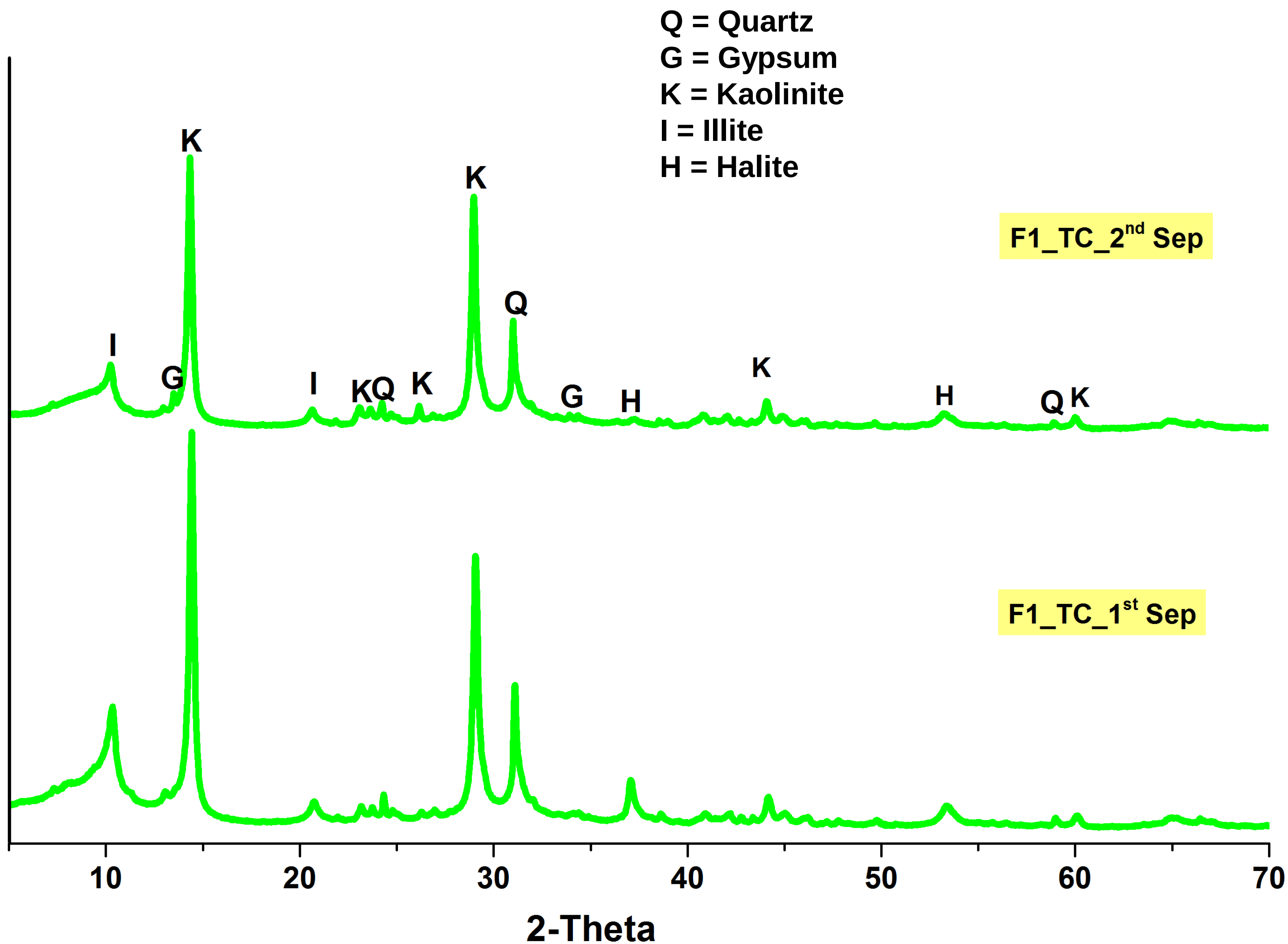
* Numbers in parenthesis refer to the last digit and represent 1σ standard uncertainty errors.

Separation of froth F1 by wettability (WET) followed by clay-ultrafines (CUF) separation

* Numbers in parenthesis refer to the last digit(s) and represent 1σ standard uncertainty errors.



XRD powder patterns measured for total clays (TC)



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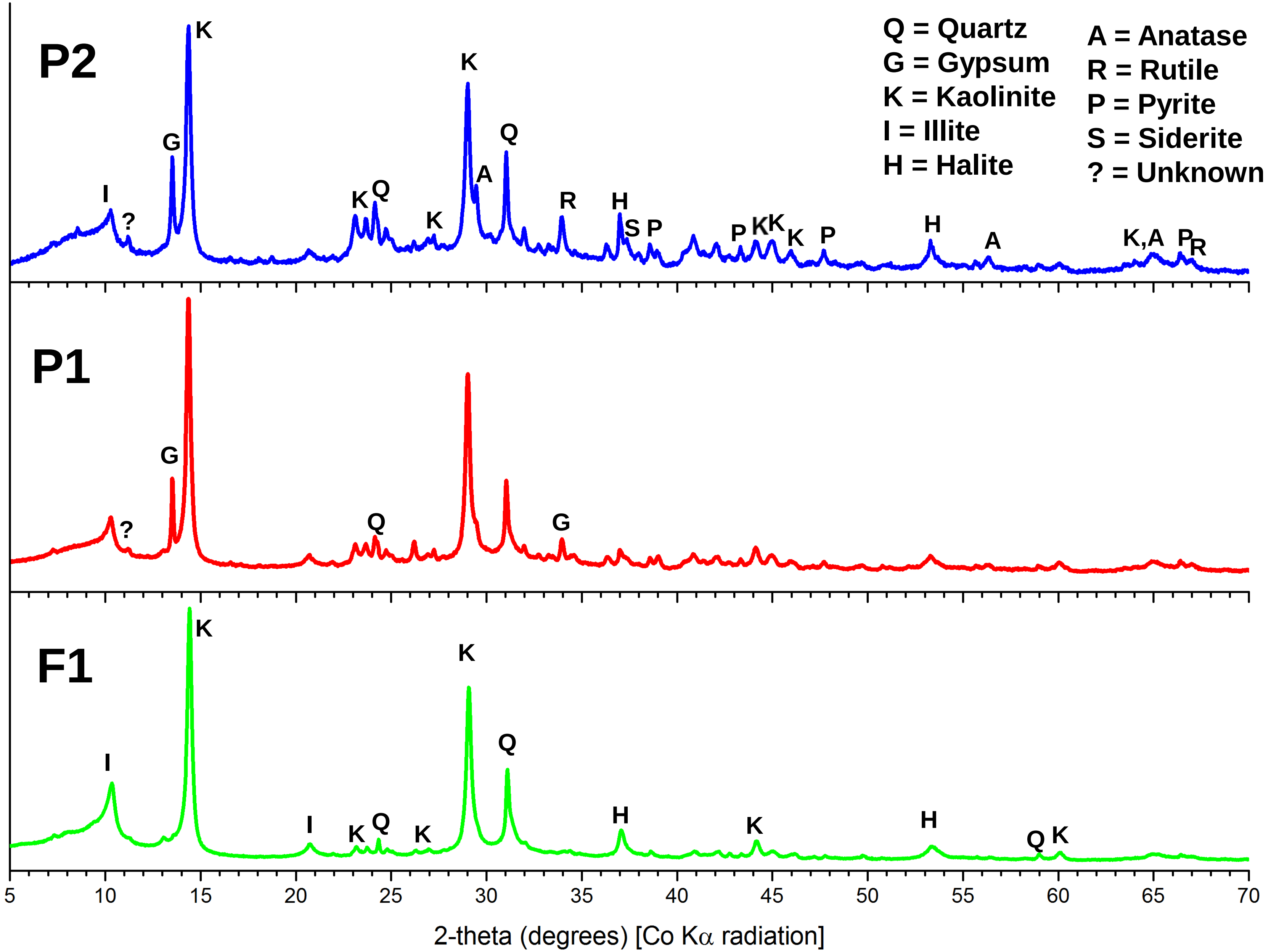

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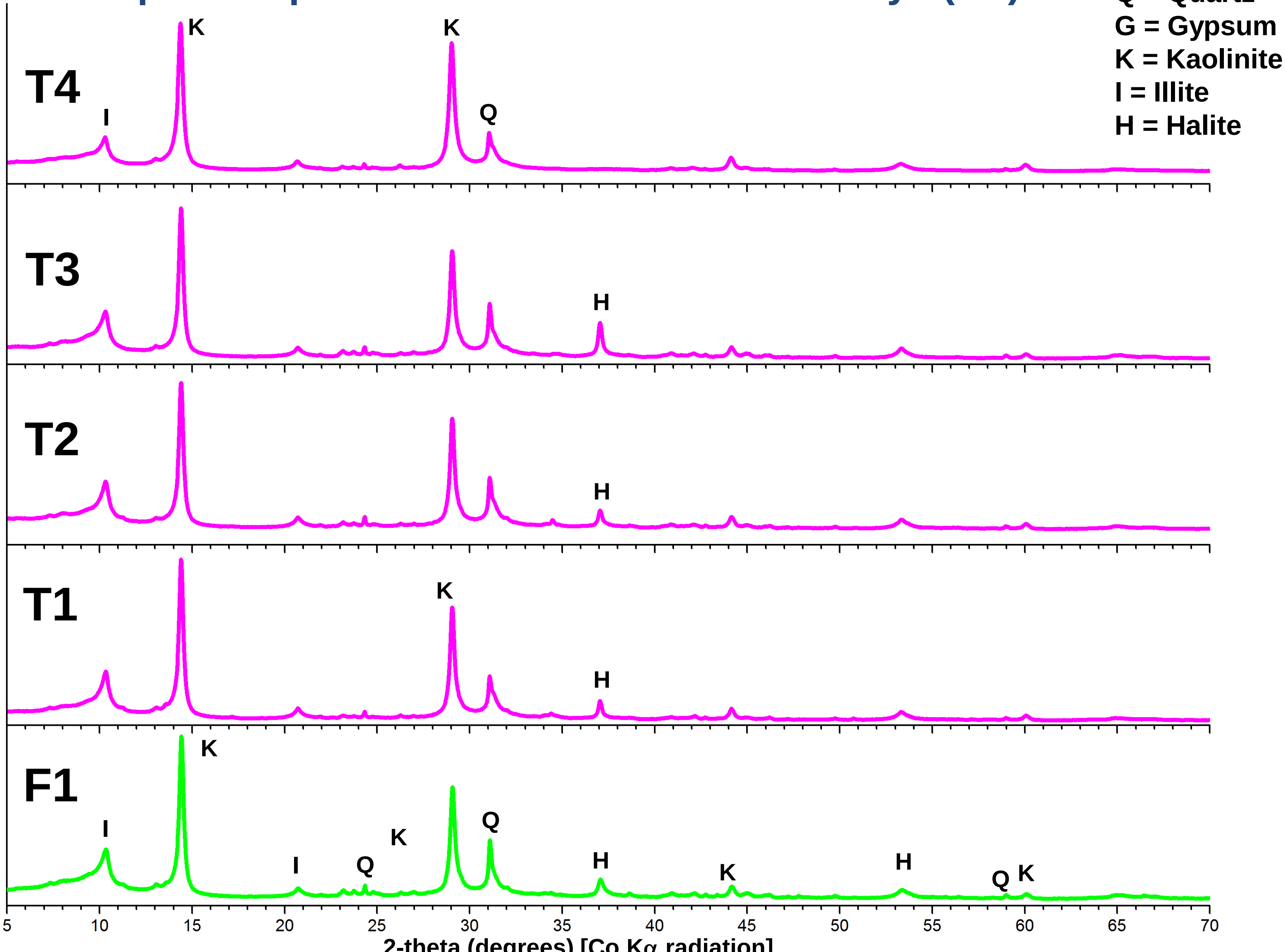
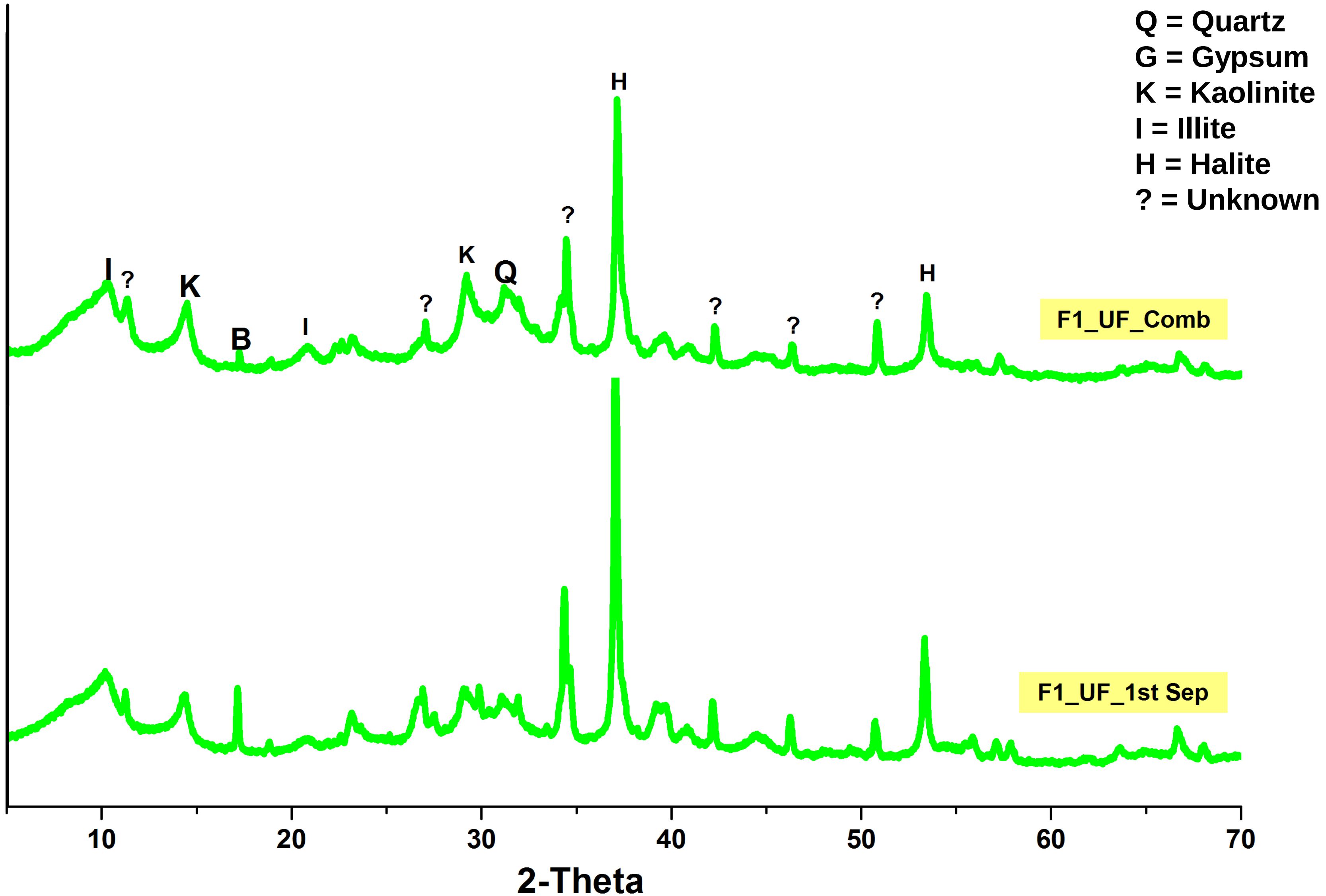


XRD powder patterns measured for ultrafines (UF)



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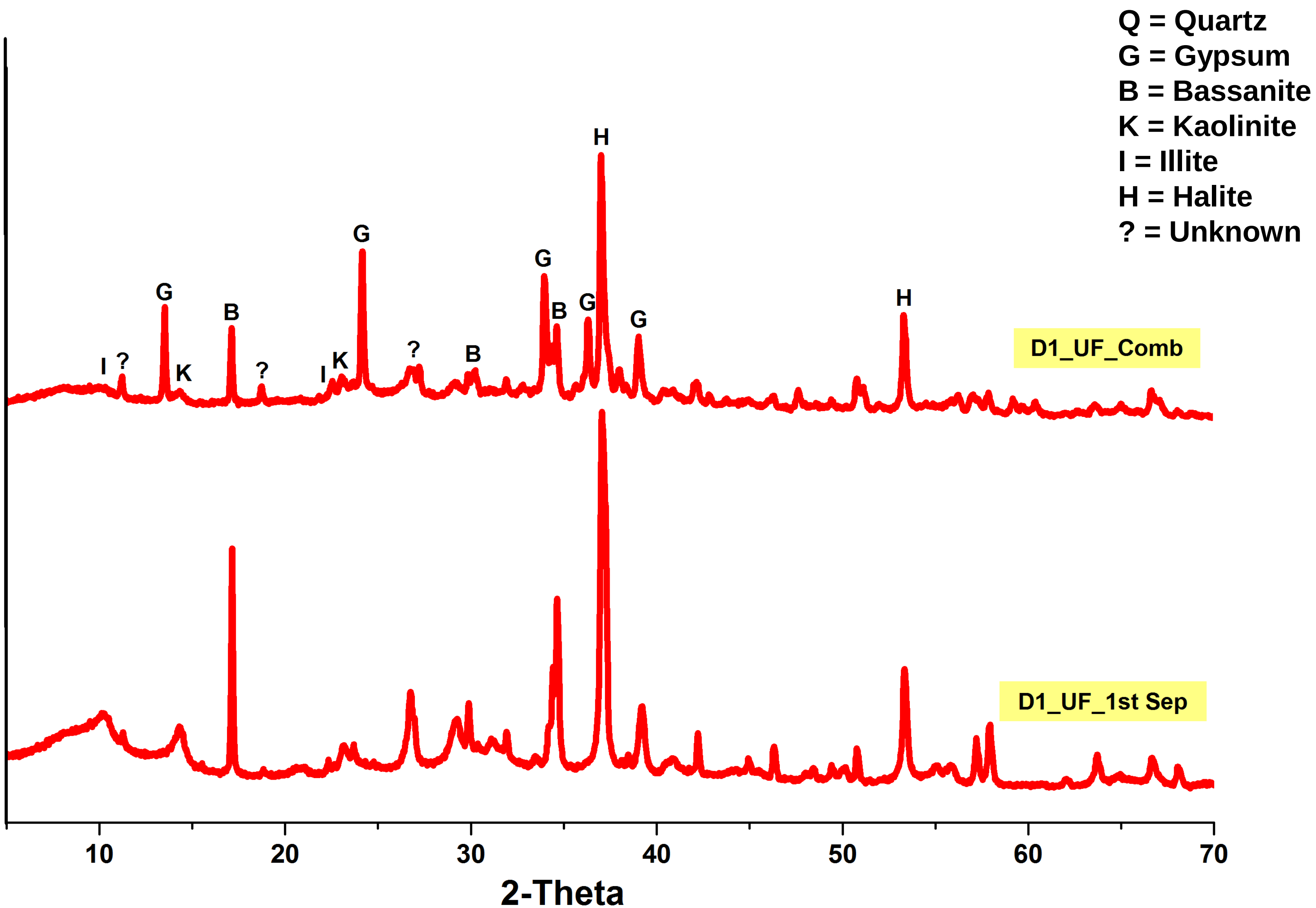
XRD powder patterns measured for ultrafines (UF)



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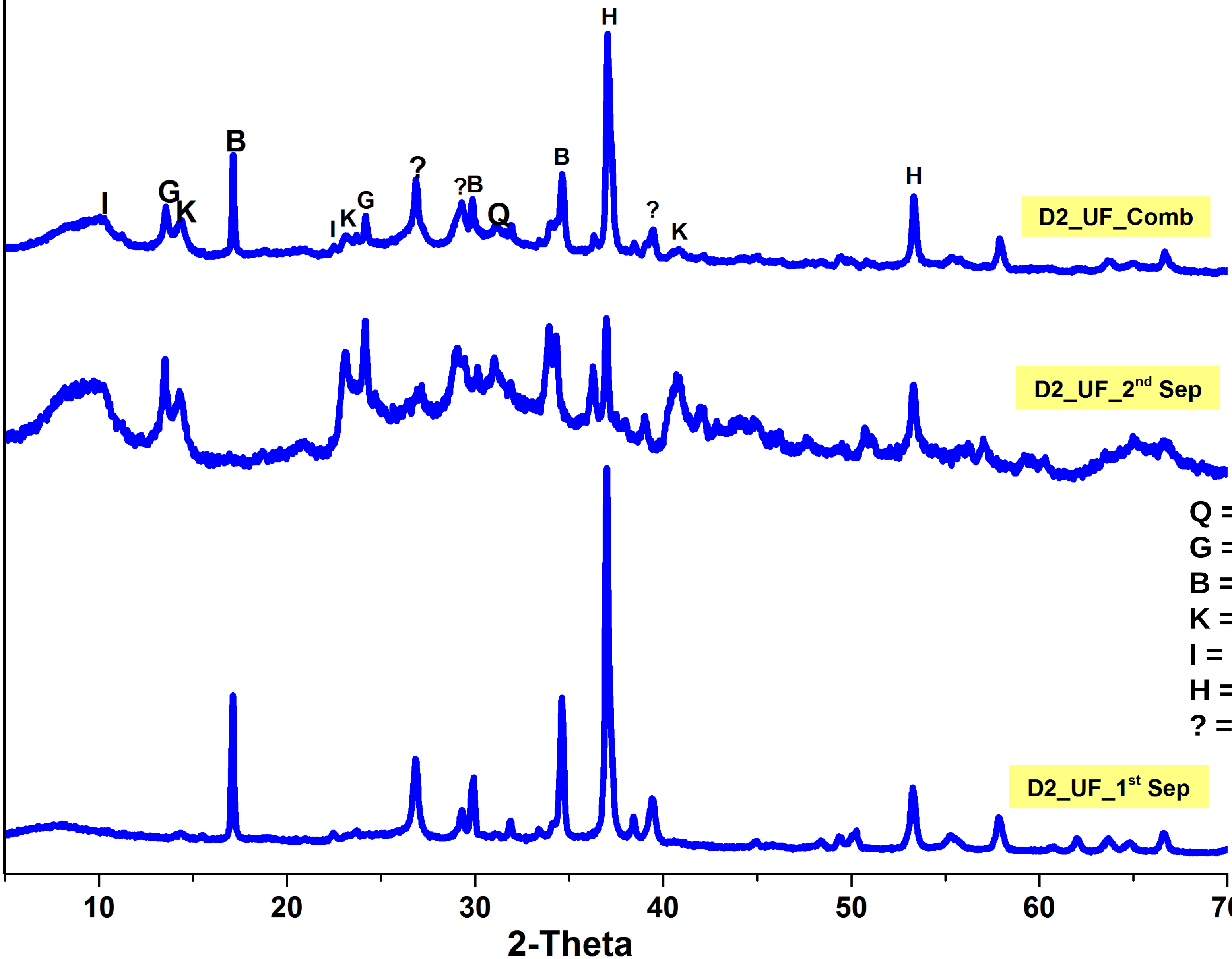
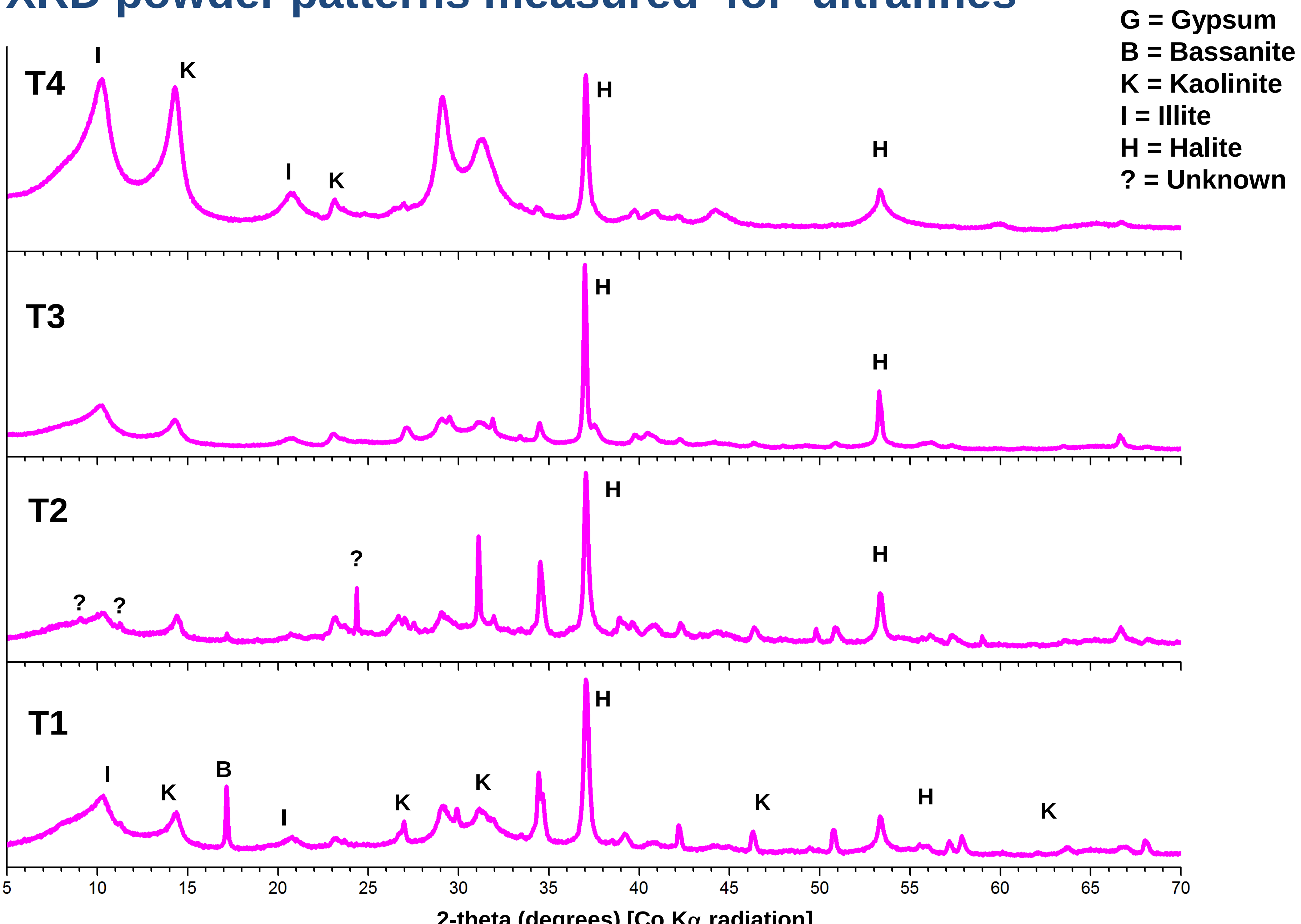
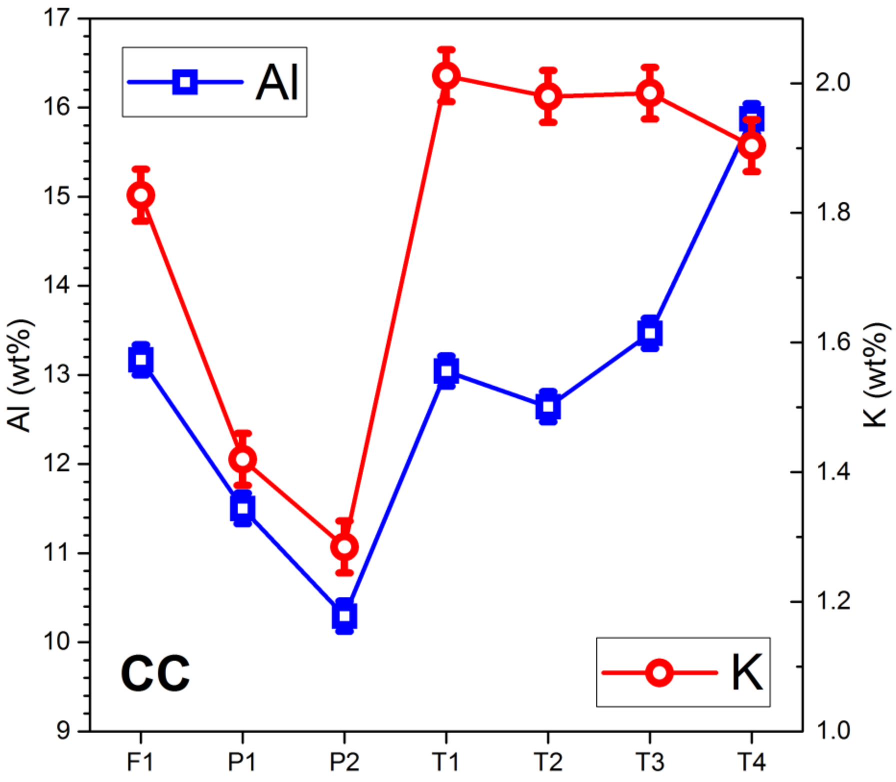


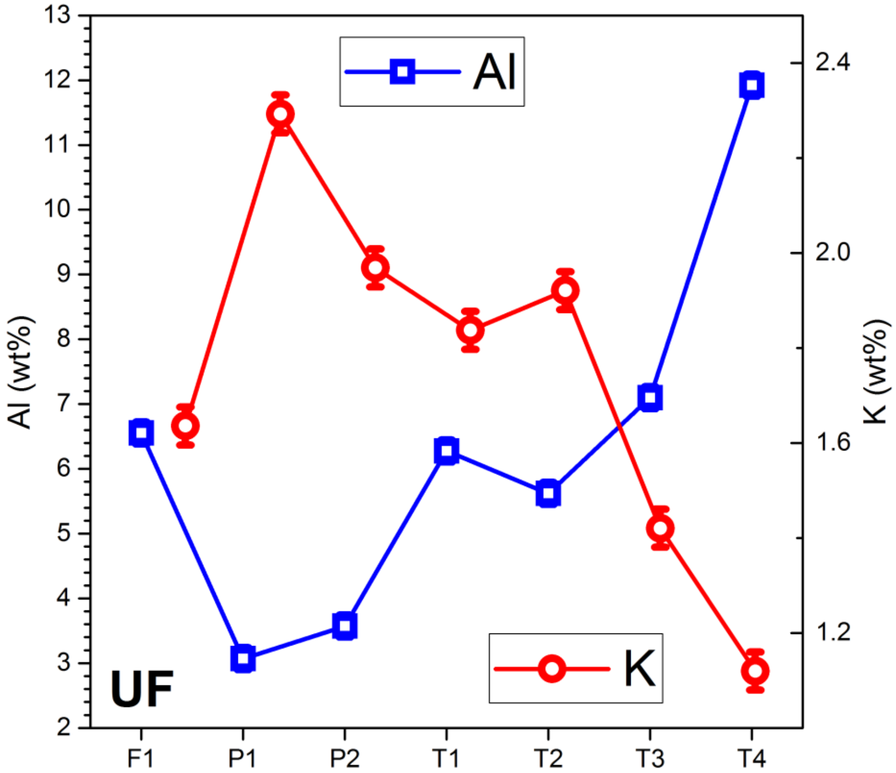
Q = Quartz
G = Gypsum
B = Bassanite
K = Kaolinite
I = Illite
H = Halite
? = Unknown

XRD powder patterns measured for ultrafines



Elemental composition results for CC and UF fractions





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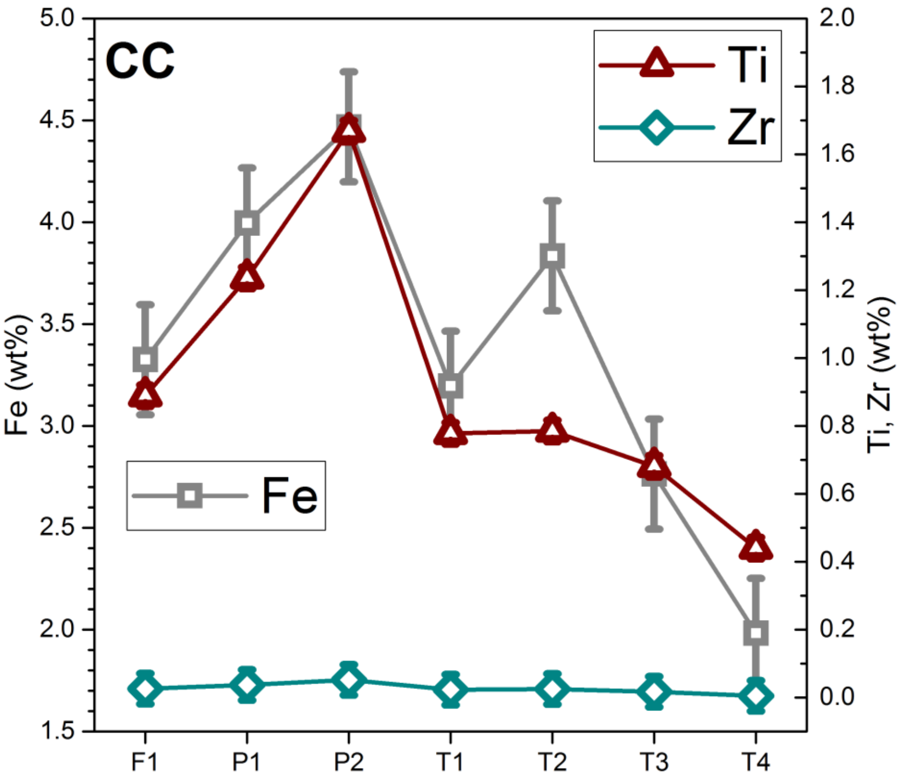

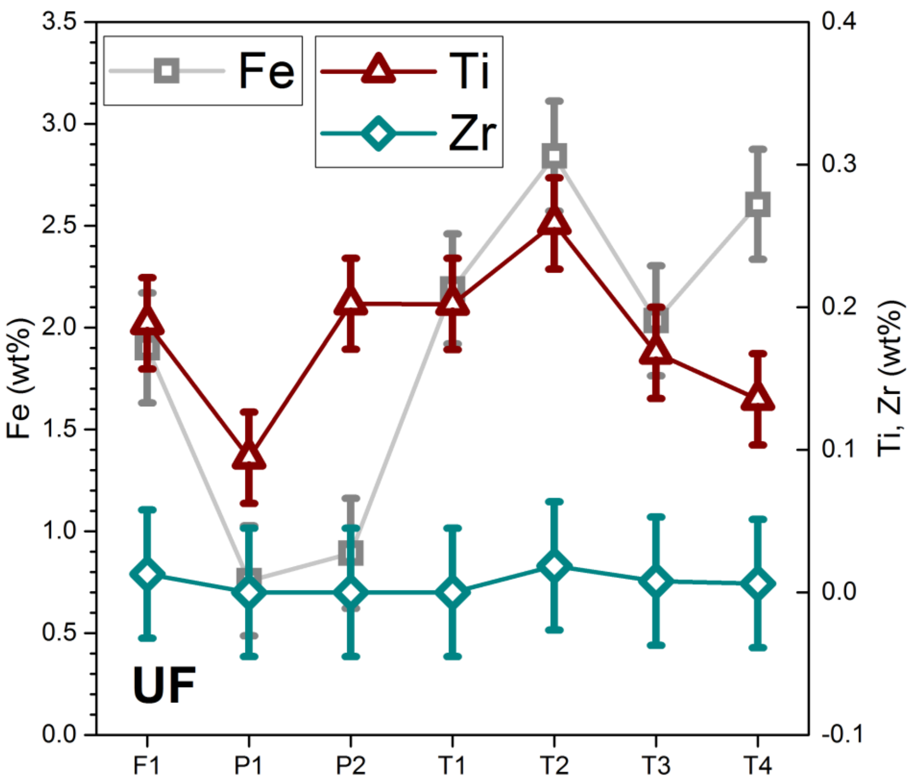

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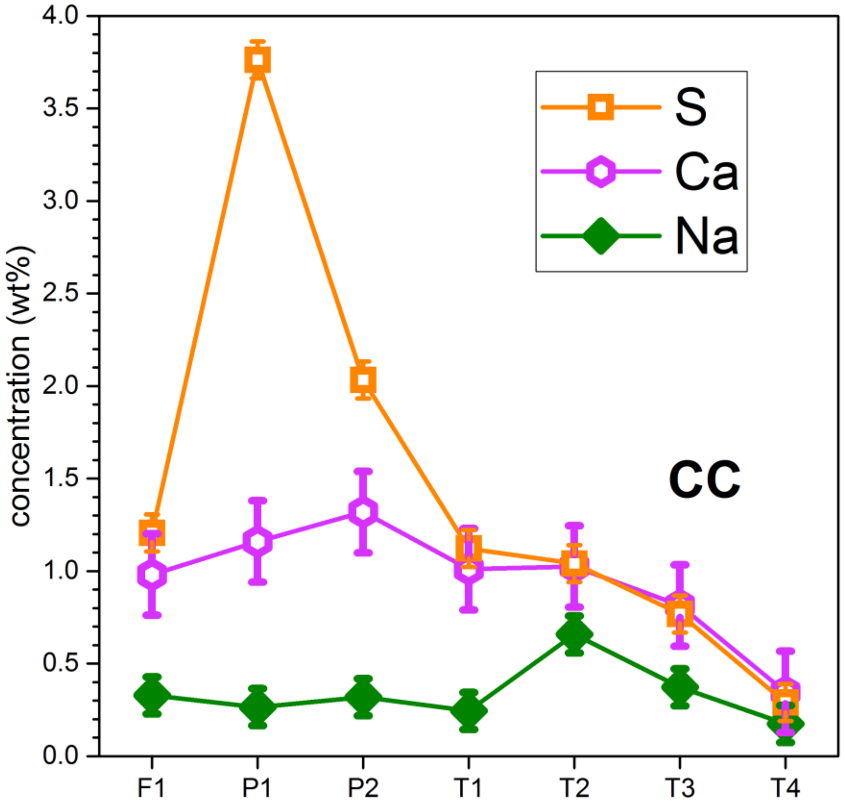

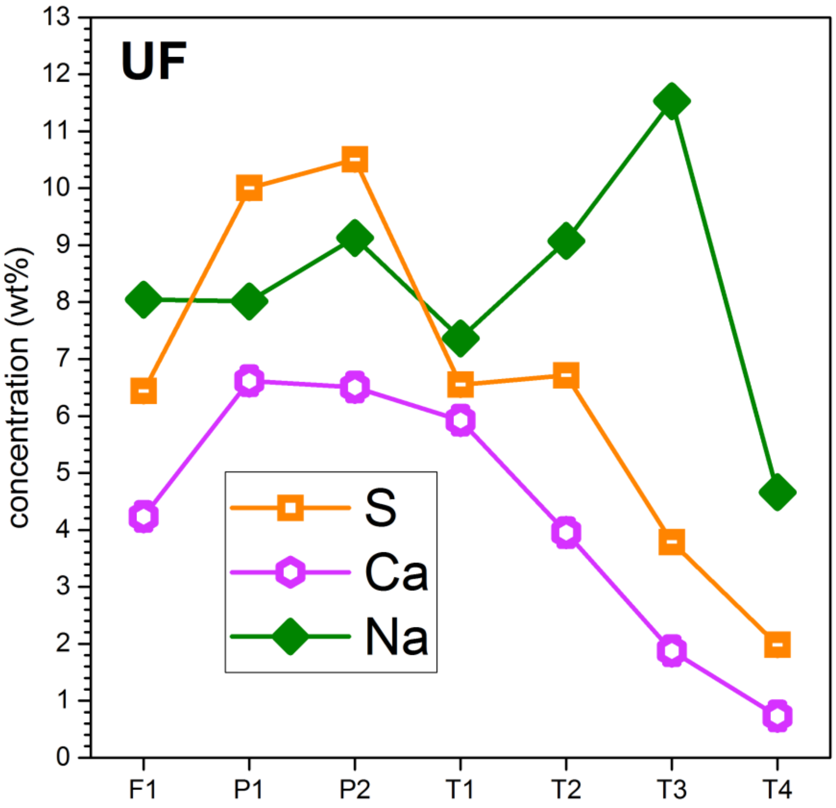

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Mineralogical compositions by methodology developed at the NRC: Singular-Value Decomposition Quantitative Phase Analysis (SVD-QPA)

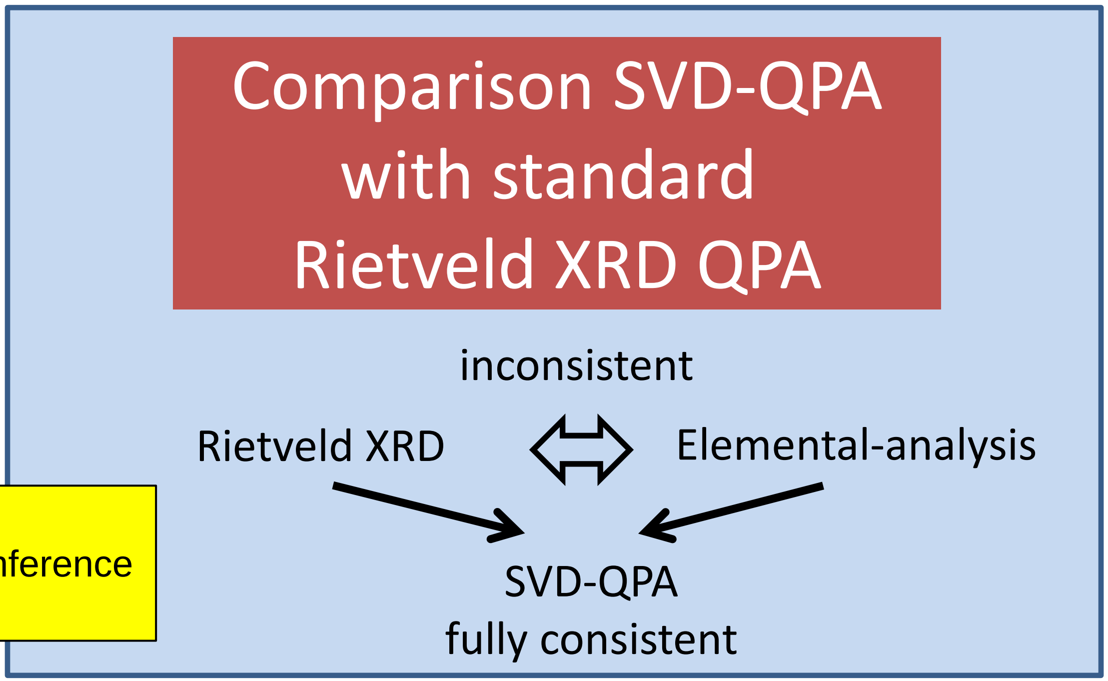
Incorporate experimental results from:

-- K, Al, Si, Fe, Ca, Mg, Ti, and Zr concentrations from XRF spectrometry

-- C and S concentrations from elemental analysis

-- mineral mass ratios of crystalline phases from XRD powder patterns

into a **single weighted linear least-squares refinement for QPA**



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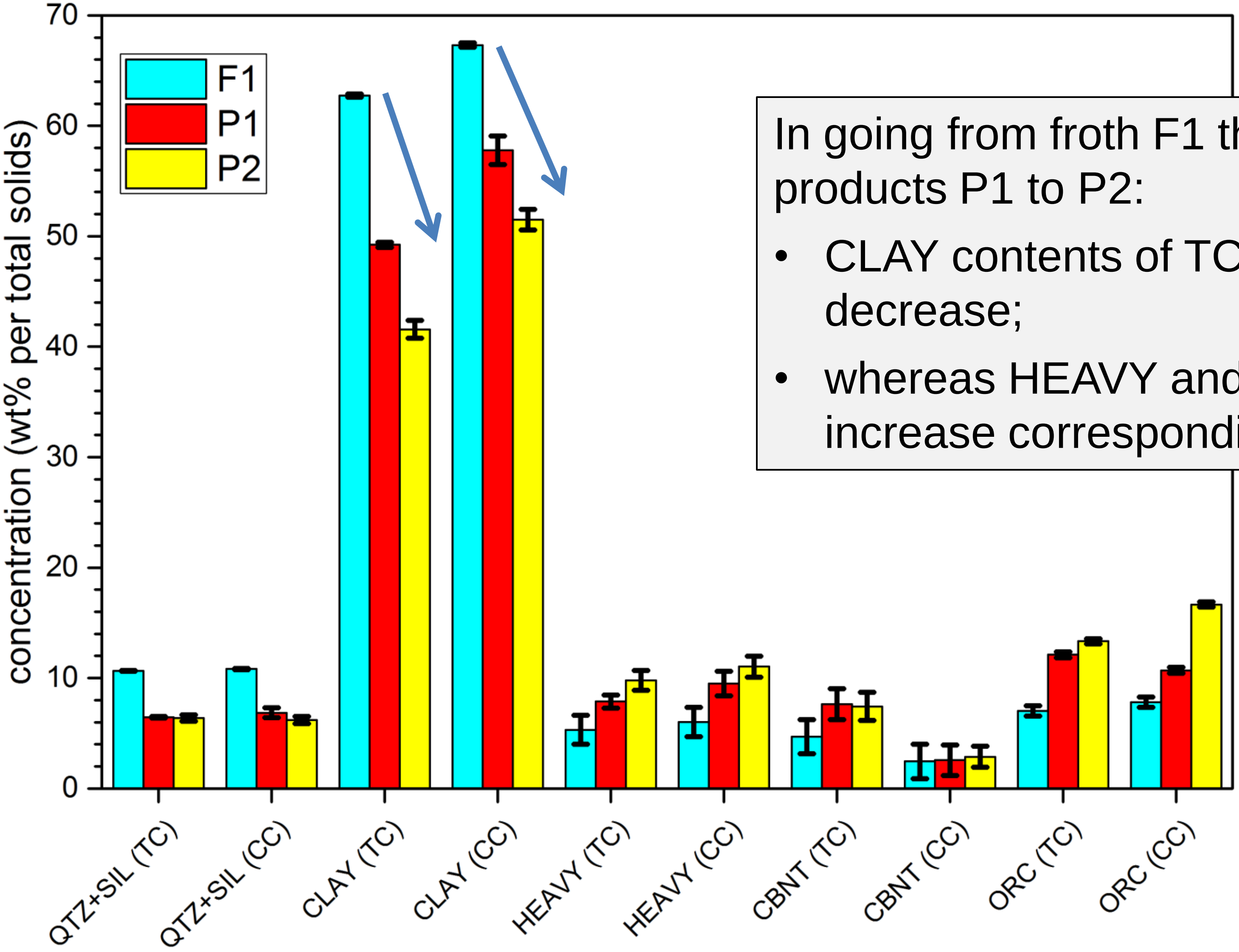
SVD-QPA mineralogical composition results for TC and CC fractions from froth and products



In going from froth F1 through products P1 to P2:

- CLAY contents of TC and CC decrease;
- whereas HEAVY and ORC increase correspondingly.

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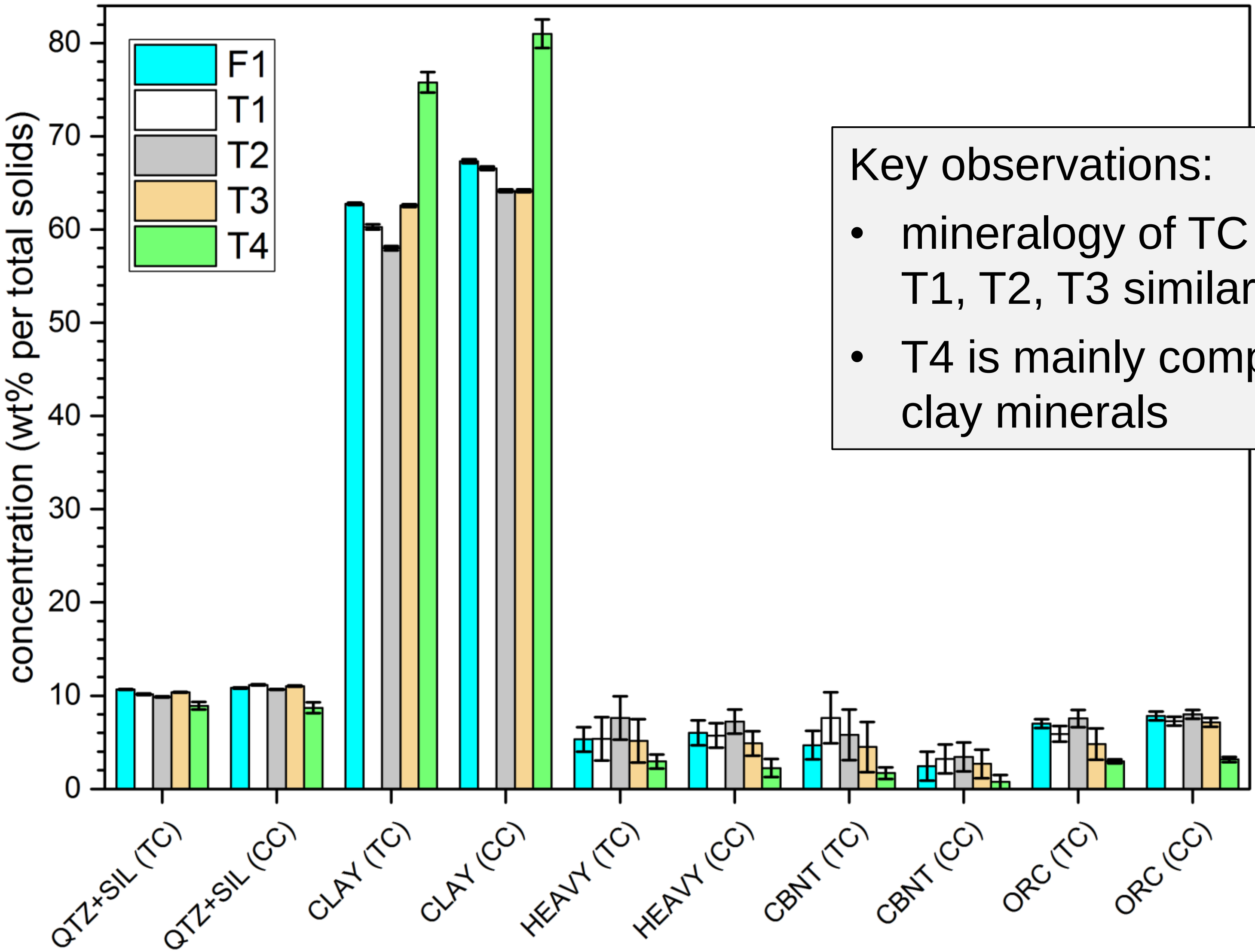
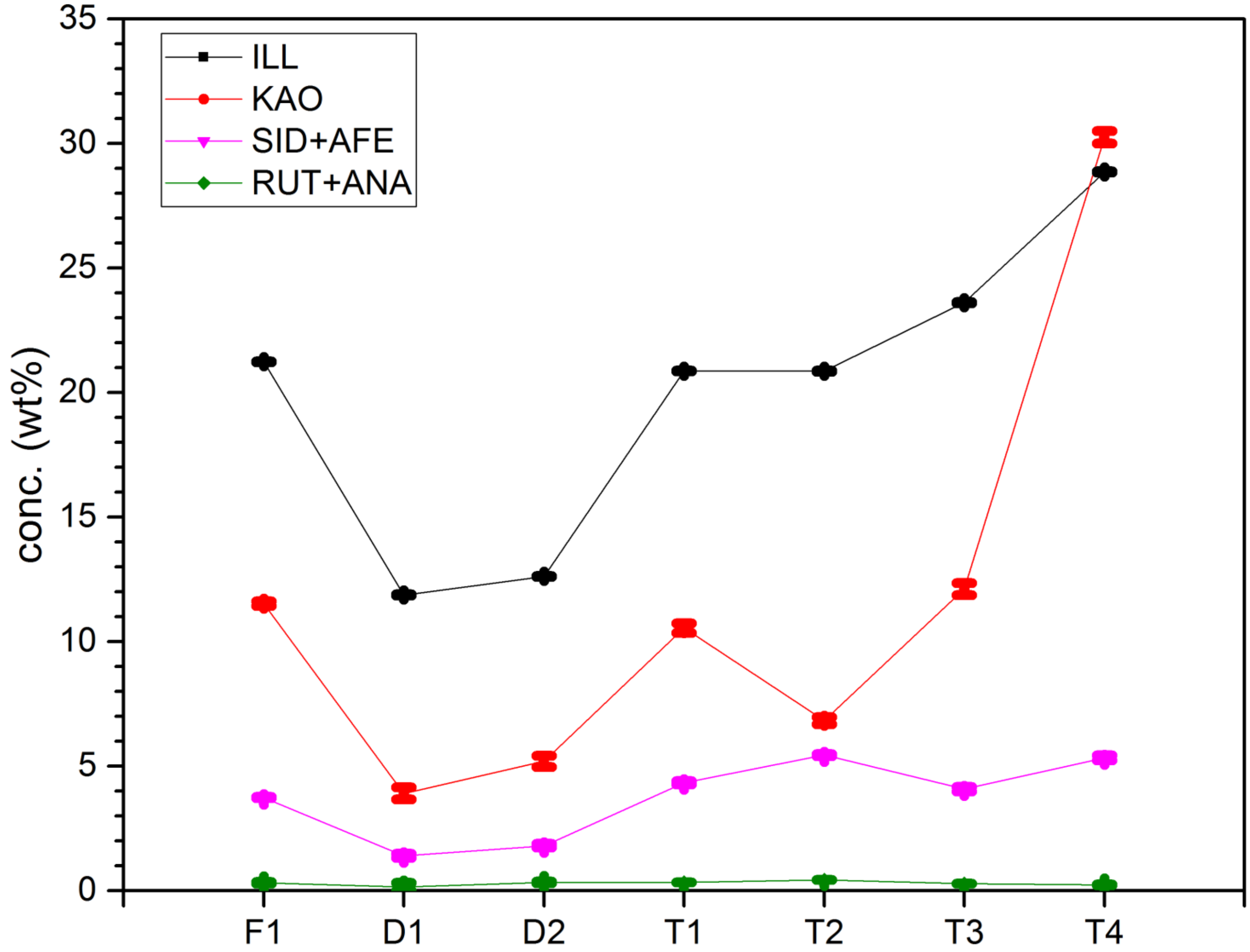
SVD-QPA mineralogical composition results for TC and CC fractions from froth and tailings



Key observations:

- mineralogy of TC and CC in T1, T2, T3 similar to F1
- T4 is mainly comprised of clay minerals

SVD-QPA mineralogical composition results for UF fractions



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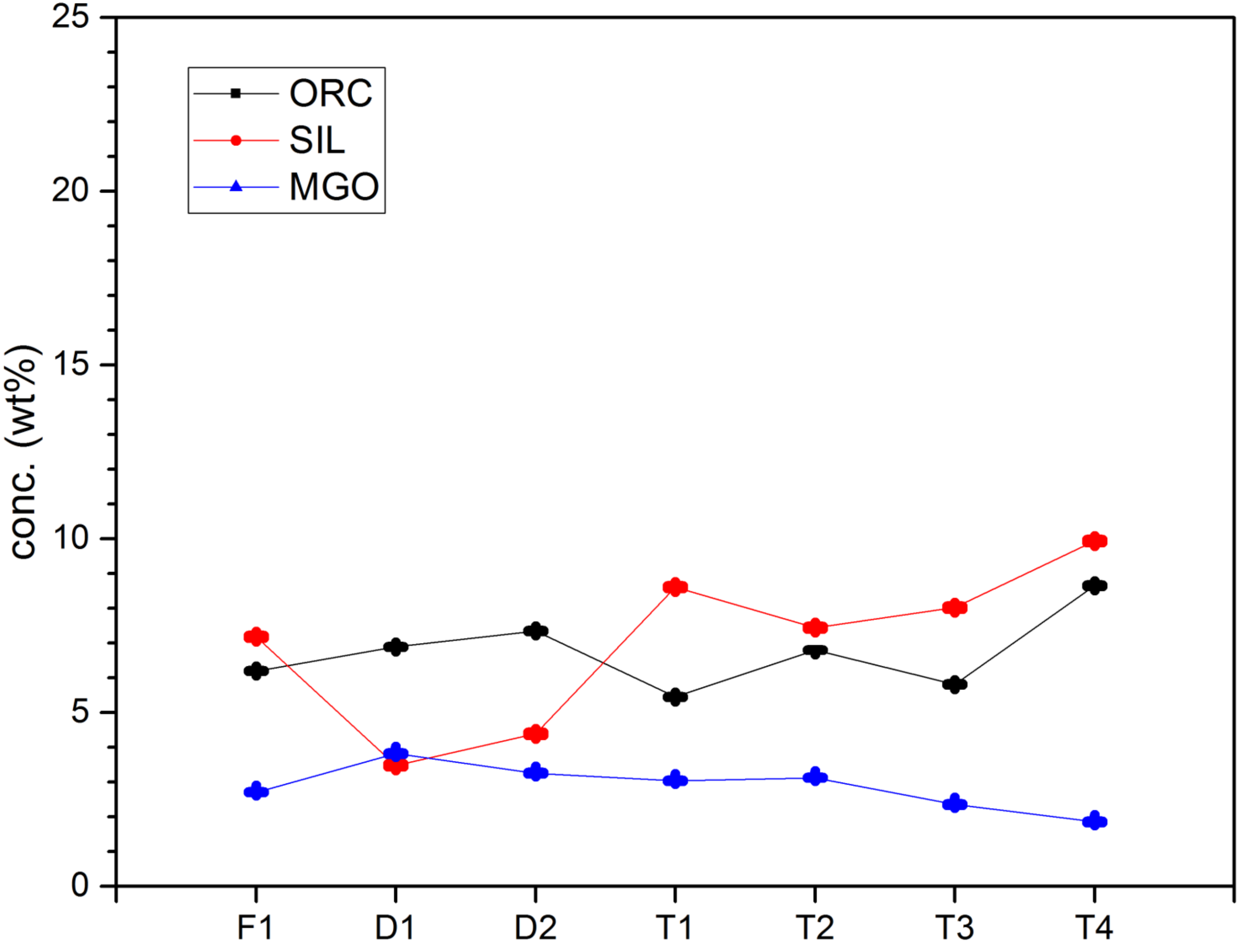

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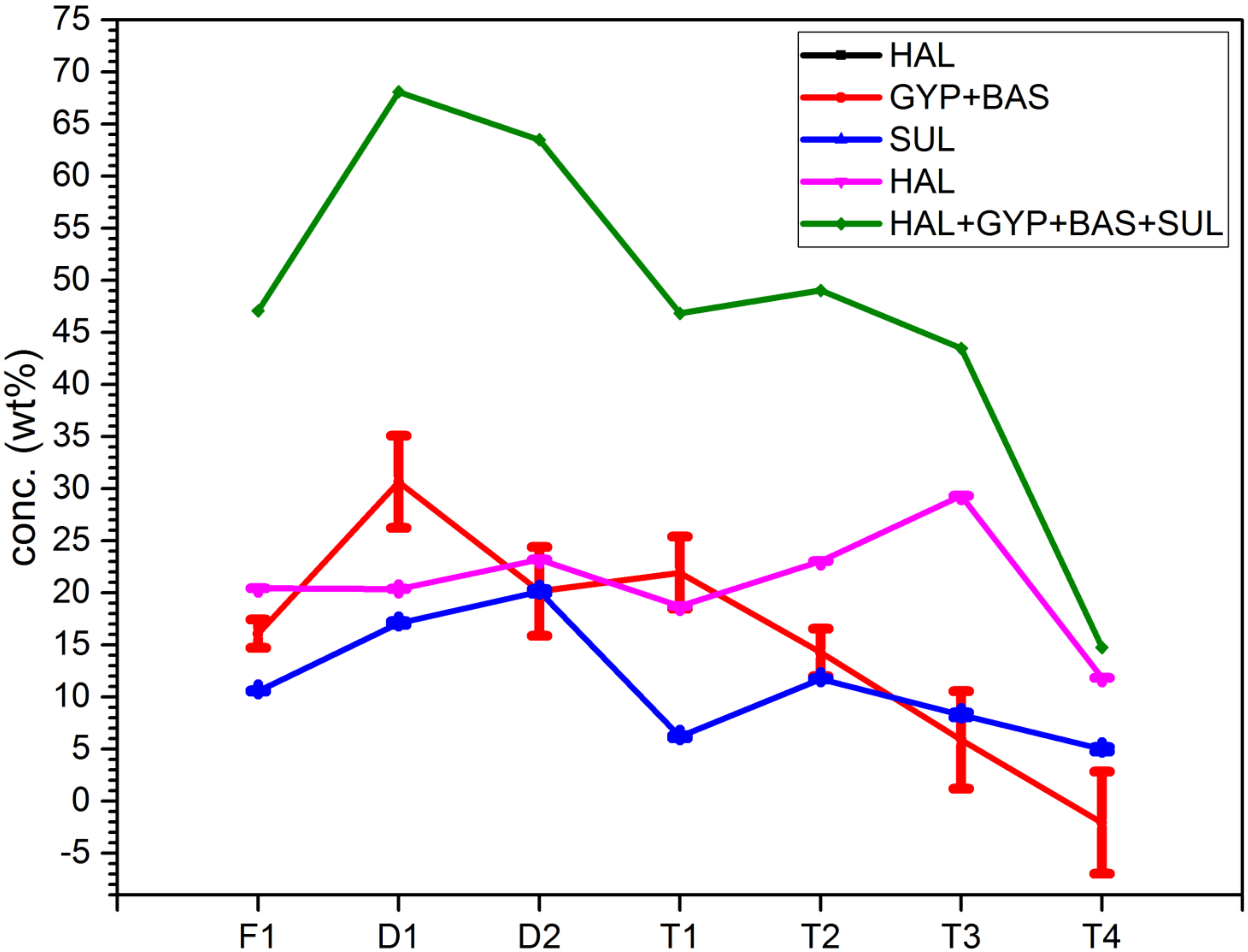
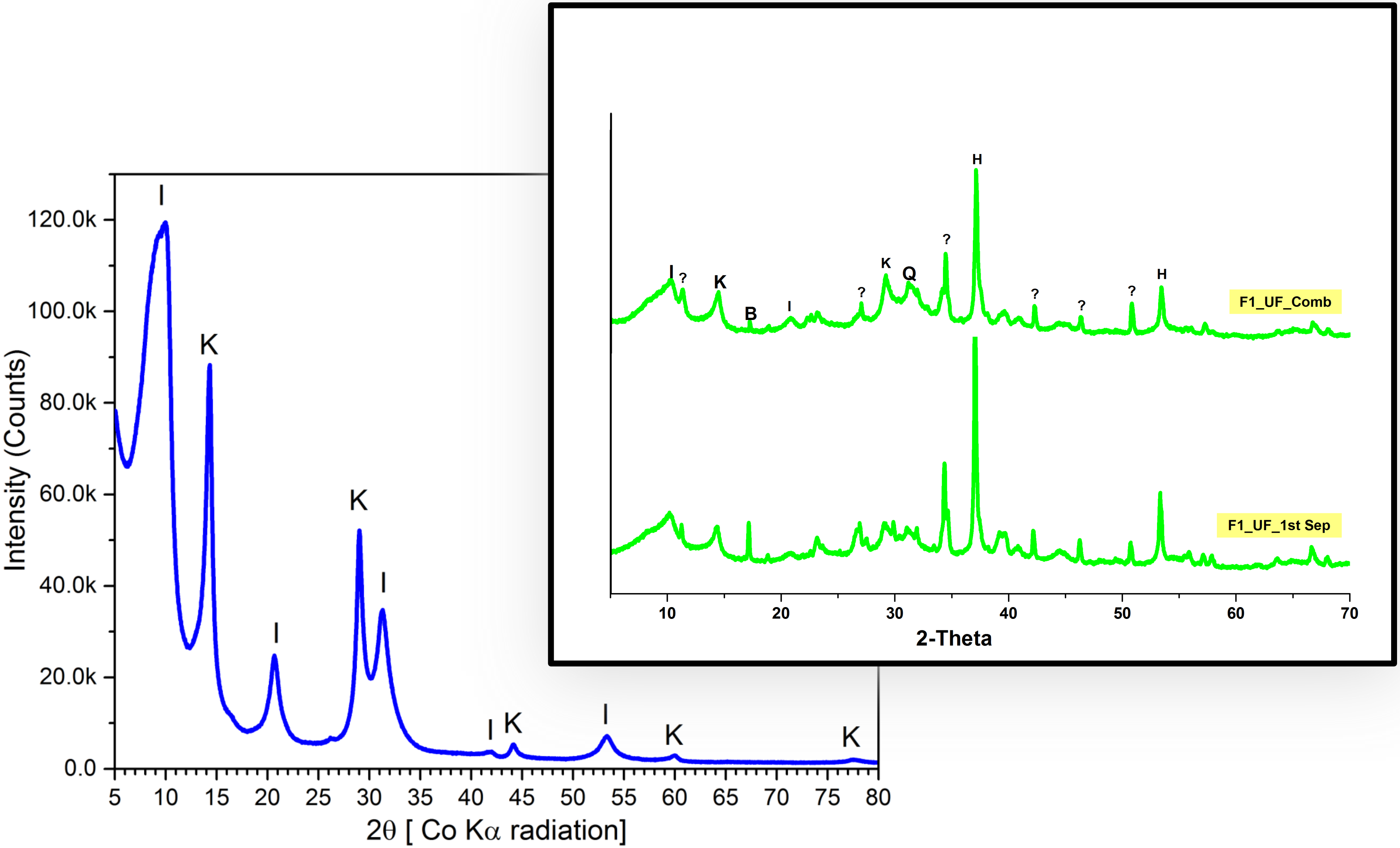


XRD pattern measured for salt-free UF fraction from froth F1 separated using modified separation technique



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Conclusions

- For the first time, the absolute amounts and compositional properties of <2μm clay and <0.2μm ultrafine solids were quantified in commercial NFT streams produced at mined oil sands operations. *Our developed technique showed >10 wt% ultrafines in samples T4, P1 and P2; whereas standard technique did not notice any significant differences between the seven samples.*
- In going from froth F1 through products P1 to P2, mineralogy in TC and CC varies as: CLAY contents decrease from ~62–68 wt% down to ~40–50 wt%, whereas HEAVY and ORC contents increase correspondingly.
- Mineralogy of TC and CC in F1 similar to T1, T2, T3: ~60–65 wt% CLAY, ~10 wt% QTZ+SIL, <10 wt% HEAVY, ~10 wt% ORC, <5 wt% CBNT.
- Mineralogy of TC and CC in T4 is markedly different: ~78–82 wt% CLAY, ~10 wt% QTZ+SIL, <5 wt% HEAVY, <5wt% ORC, <5 wt% CBNT.
- Higher concentrations of Na, Ca and S occurred in the UF solids compared to corresponding TC and CC fractions, and many unknown peaks were present in XRD patterns of UF fractions.

  Using a slightly modified separation procedure, which prevented water to be dried along with UF solids upon isolating the latter, we were able to isolate a UF fraction from froth F1 with XRD pattern showing that all diffraction peaks belong to either illite or kaolinite.
  => This suggests that soluble salts are present in the water phase within the original samples.

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Questions, comments, discussion?

Thank you

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