



nZVI PRODUCTION BY WET MILLING THROUGH THE USE OF ALUMINA

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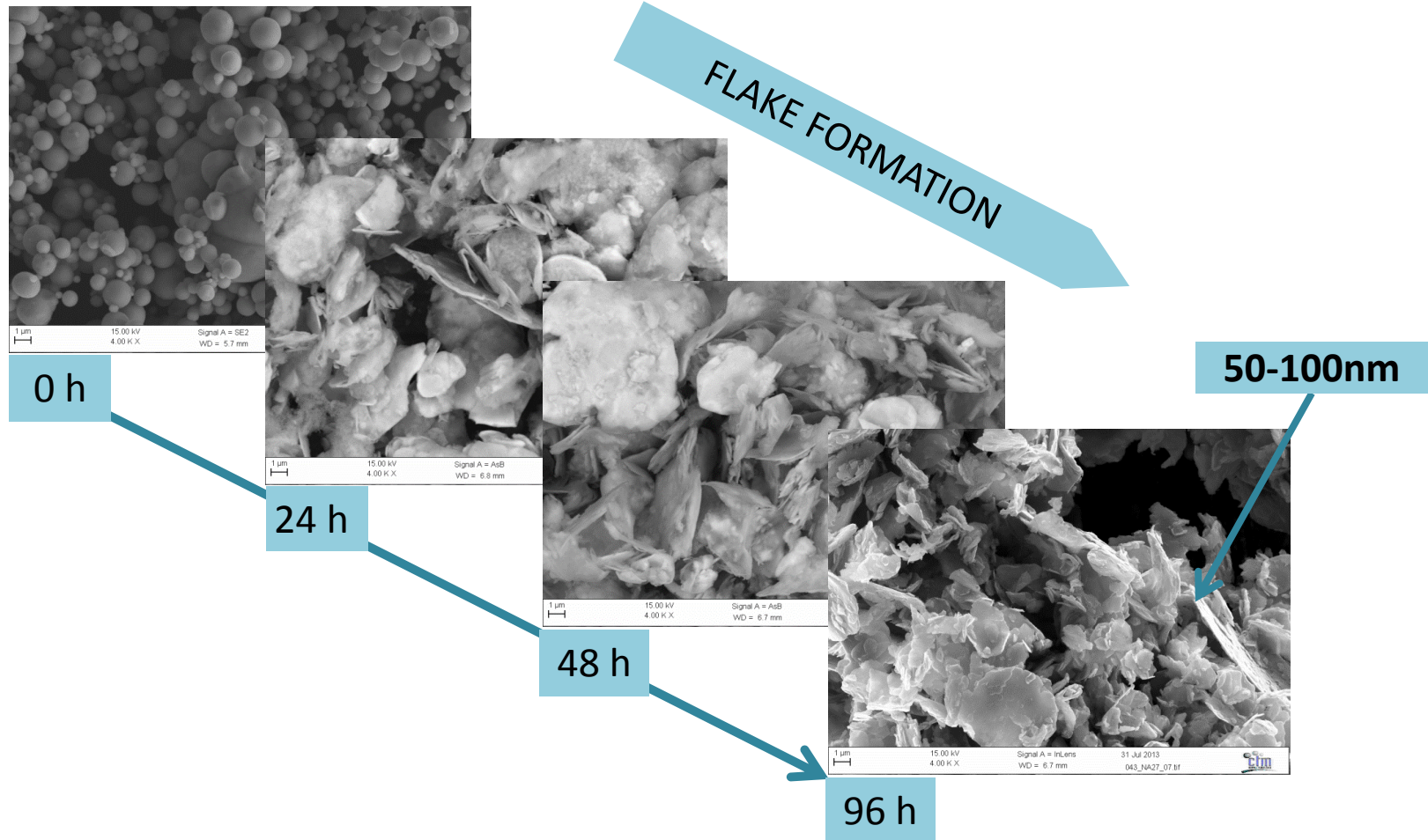
- INTRODUCTION, PREVIOUS APPROACH
- NEW RESEARCH, ABRASIVES
- TUNING THE NEW PROCESS
- GENERAL CHARACTERIZATION
- REACTIVITY CHARACTERIZATION

Introduction

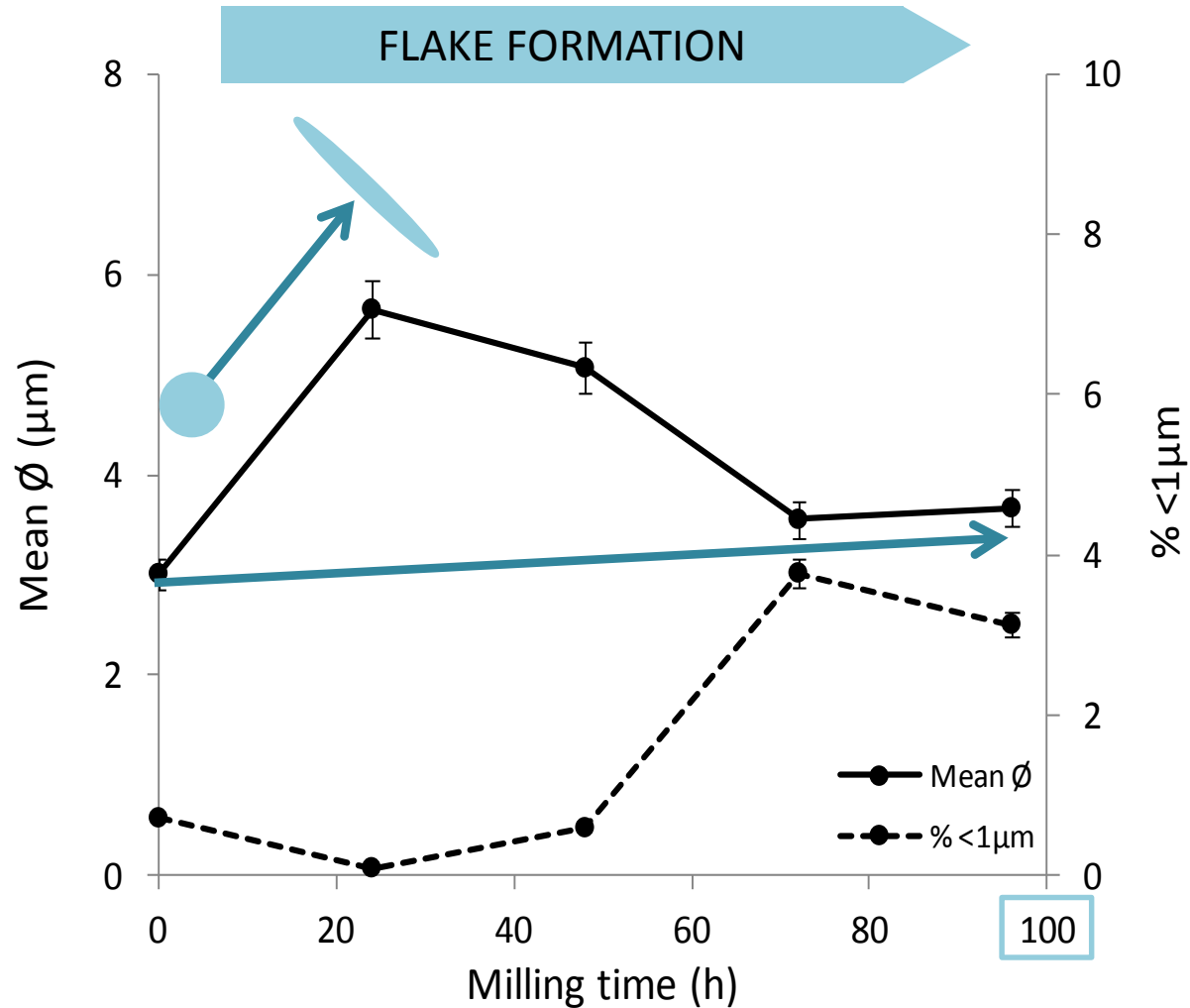
- Need for a cheaper-straightforward nZVI production method
- Top → Down approach: Wet milling under MEG
- Milling: ball milling
 - Lab scale: planetary
 - Industrial: gravitational

Introduction: Previous Approach

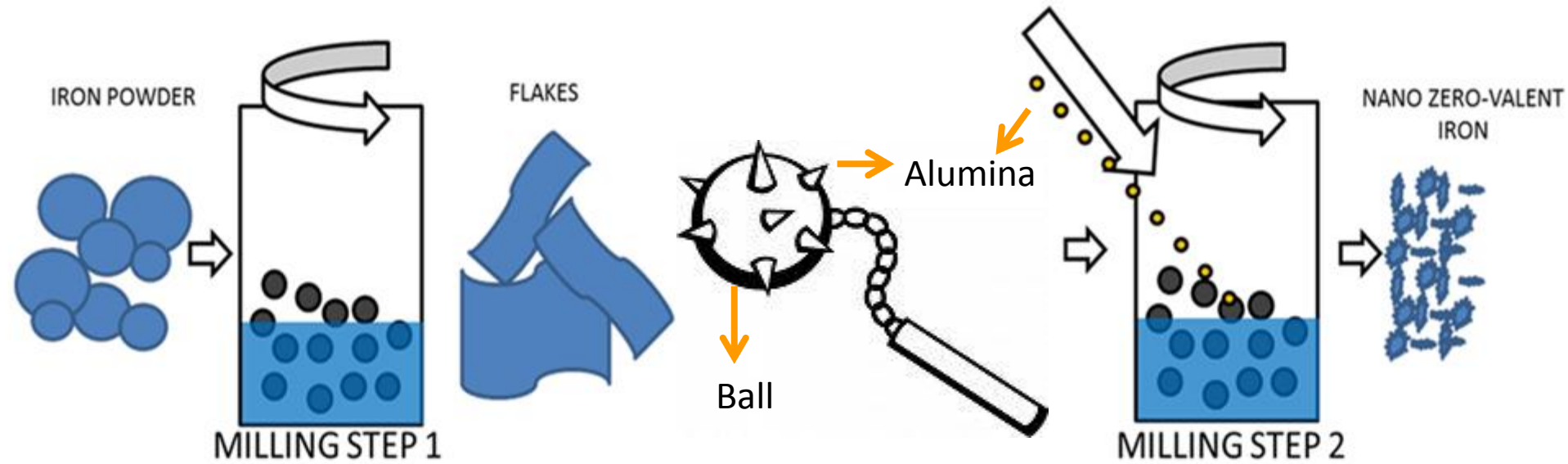
Present and previous projects stated the challenge:
iron ductility, flakes



Introduction: Previous Approach



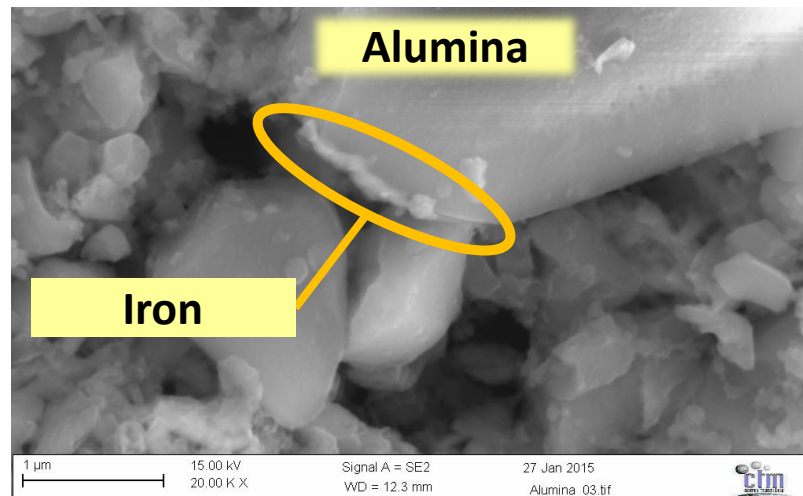
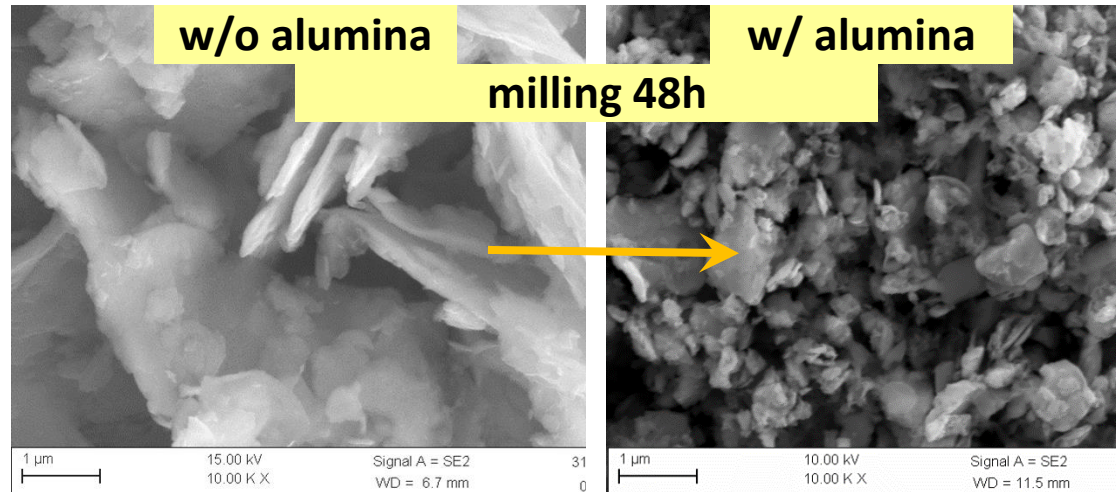
New Research, Abrasives



New Research, Abrasives

Breakage

Abrasion



Abrasives: Tuning the Process

| Ball name | Ball \emptyset mm | [Alumina] (g·l ⁻¹) | Laser Diffraction (by vol.) | |
|-----------|------------------------|-----------------------------------|-----------------------------------|------------------------|
| | | | Mean \emptyset μm | < 1 μm % |

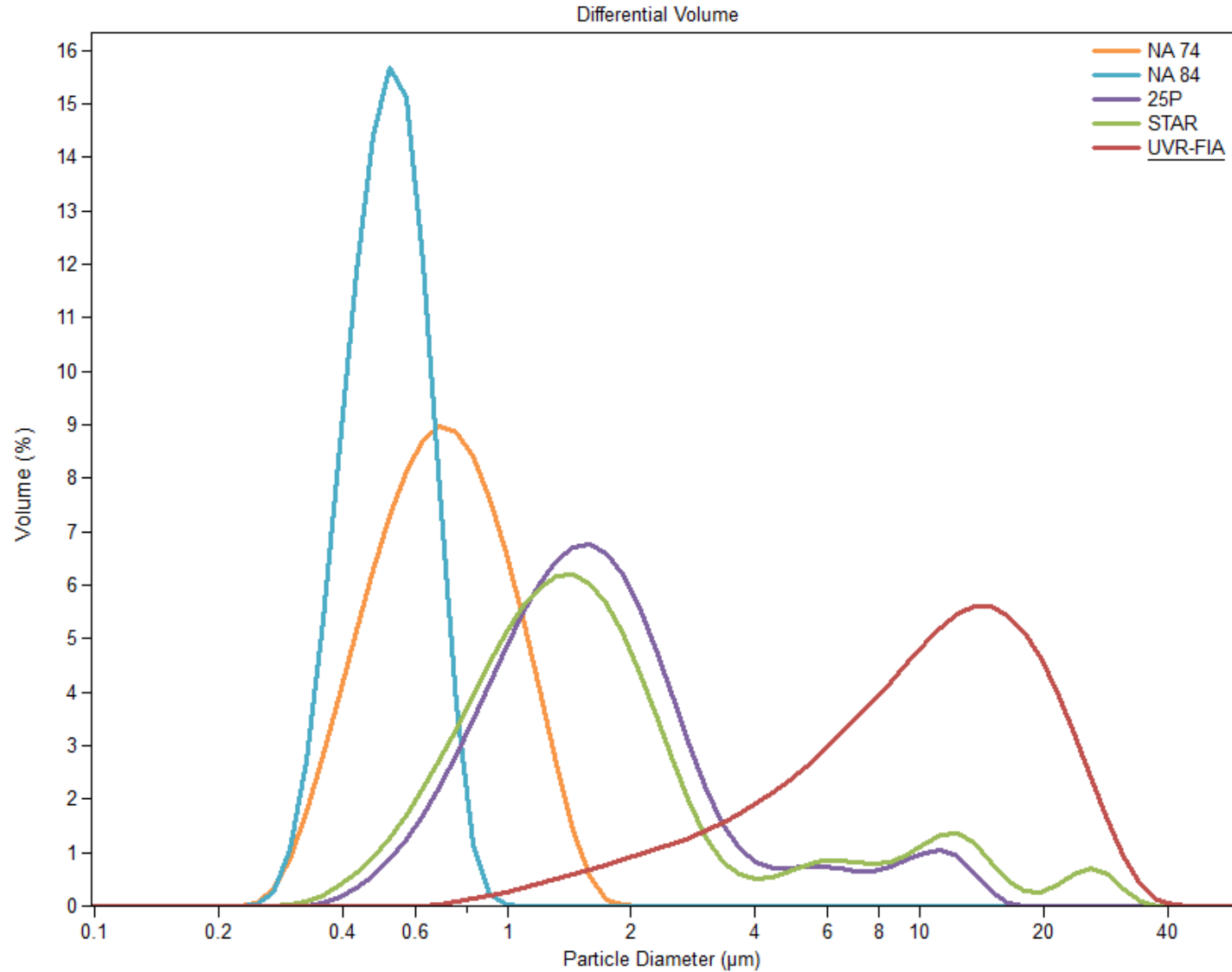
< 1 μm % vs different \emptyset grinding media

| | | | | | |
|-------|---------|------|------|------|------|
| NA 78 | MT500 | 0.1 | 53.6 | 1.13 | 38.3 |
| NA 74 | S110 | 0.5 | 53.6 | 0.76 | 76.3 |
| NA 62 | S330 | 1.05 | 53.6 | 1.19 | 48.6 |
| NA 56 | S660 | 1.7 | 53.6 | 1.12 | 41.3 |
| NA 65 | S660 | 1.7 | 53.6 | 1.16 | 40.5 |
| NA 66 | S660 | 1.7 | 53.6 | 1.28 | 33.6 |
| NA 64 | 1010 5 | 5 | 53.6 | 1.01 | 53.6 |
| NA 67 | 1010 10 | 10 | 53.6 | 1.90 | 34.5 |

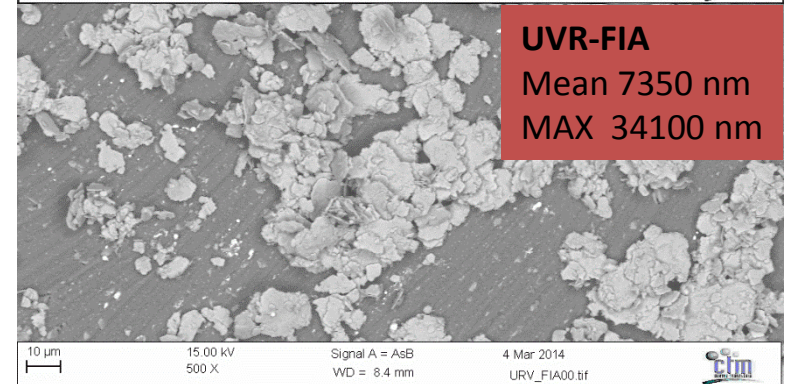
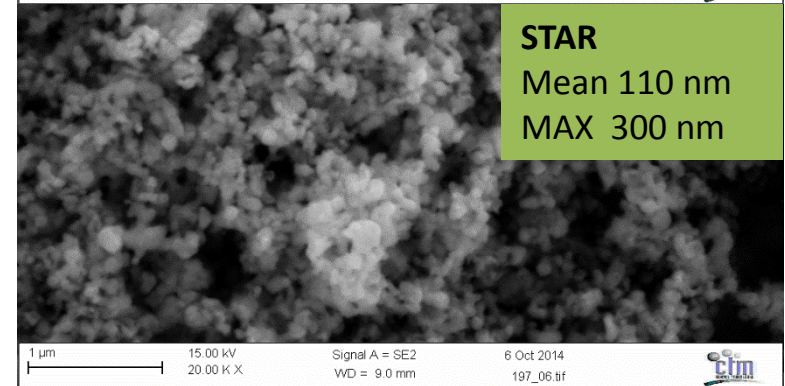
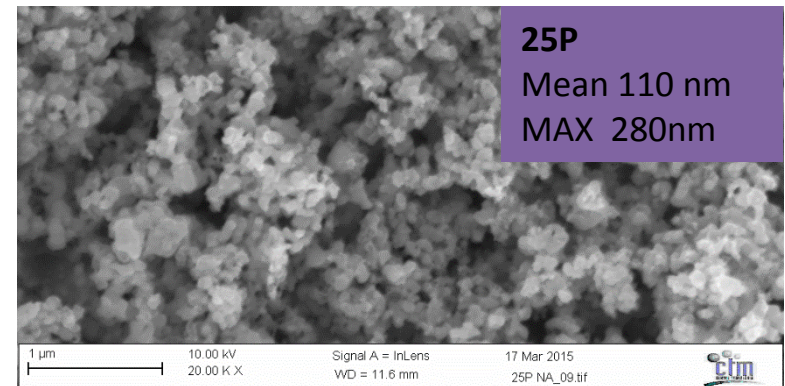
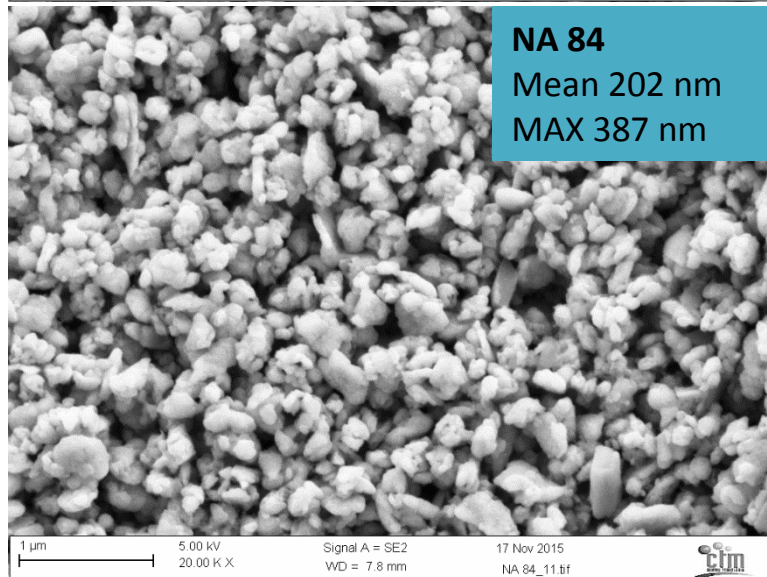
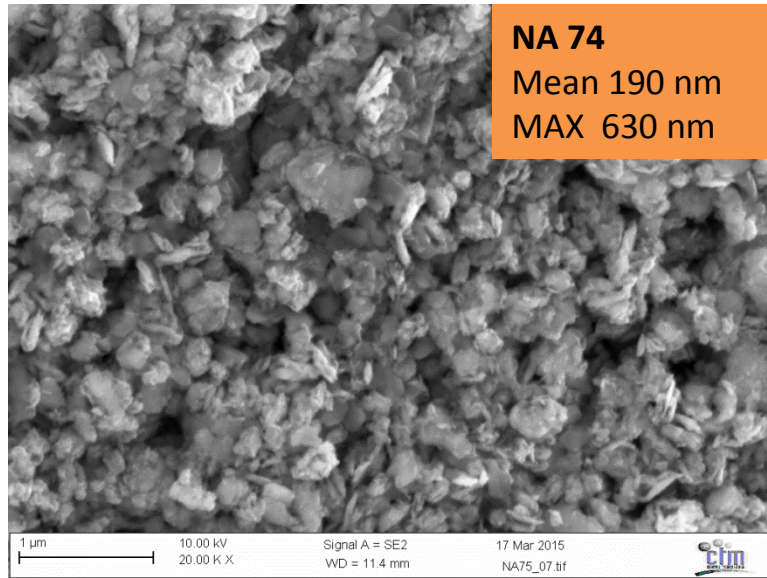
< 1 μm % vs different alumina concentration

| | | | | | |
|-------|------|-----|-------|------|------|
| NA 74 | S110 | 0.5 | 53.6 | 0.76 | 76.3 |
| NA 84 | S110 | 0.5 | 80.4 | 0.52 | 100 |
| NA 83 | S110 | 0.5 | 107.2 | 0.93 | 56.8 |

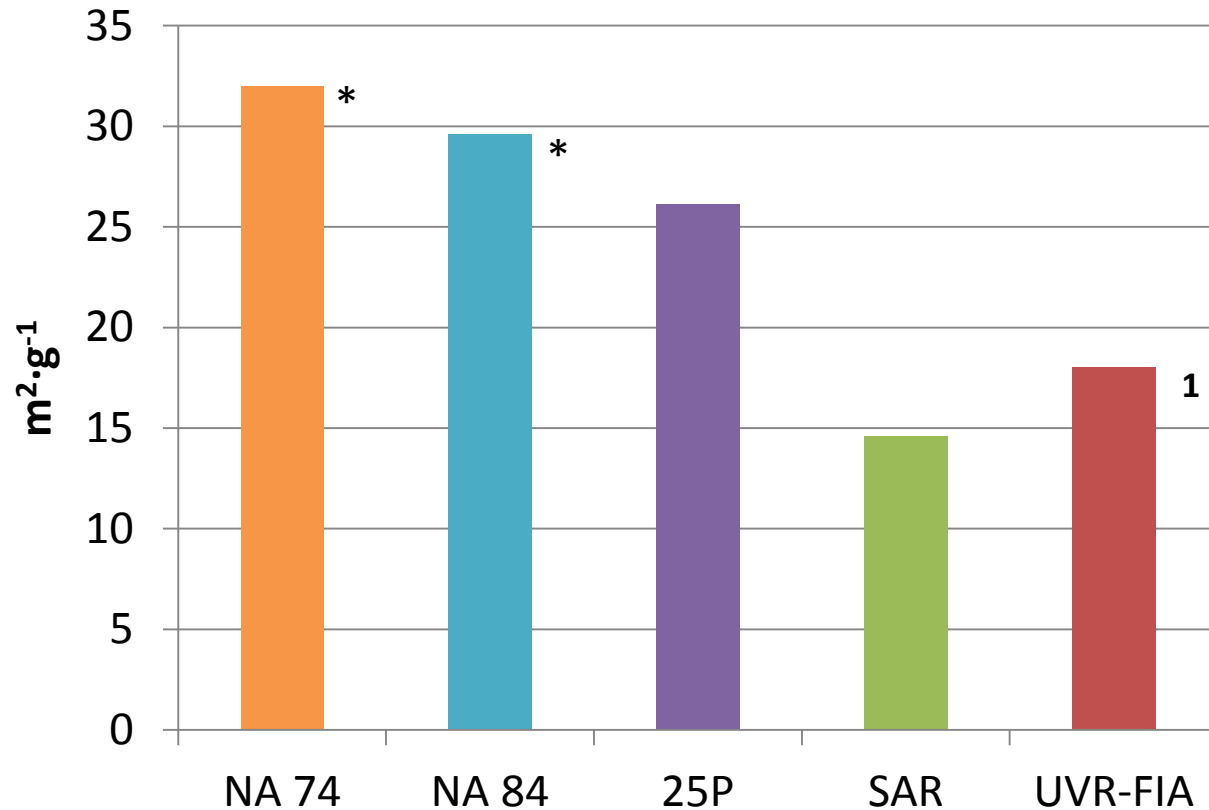
Size characterization: LD



Size characterization: SEM



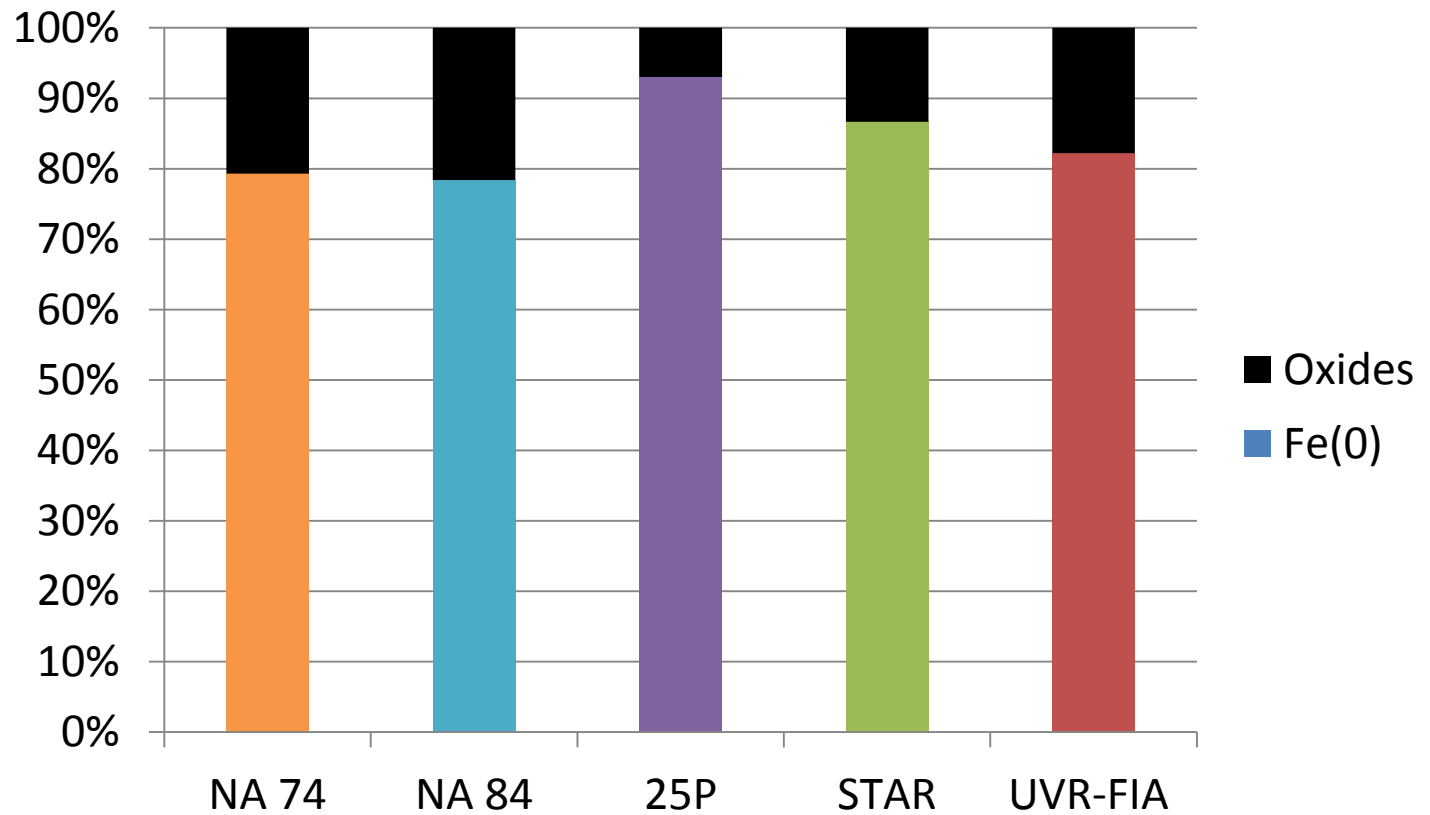
Size Characterization: BET



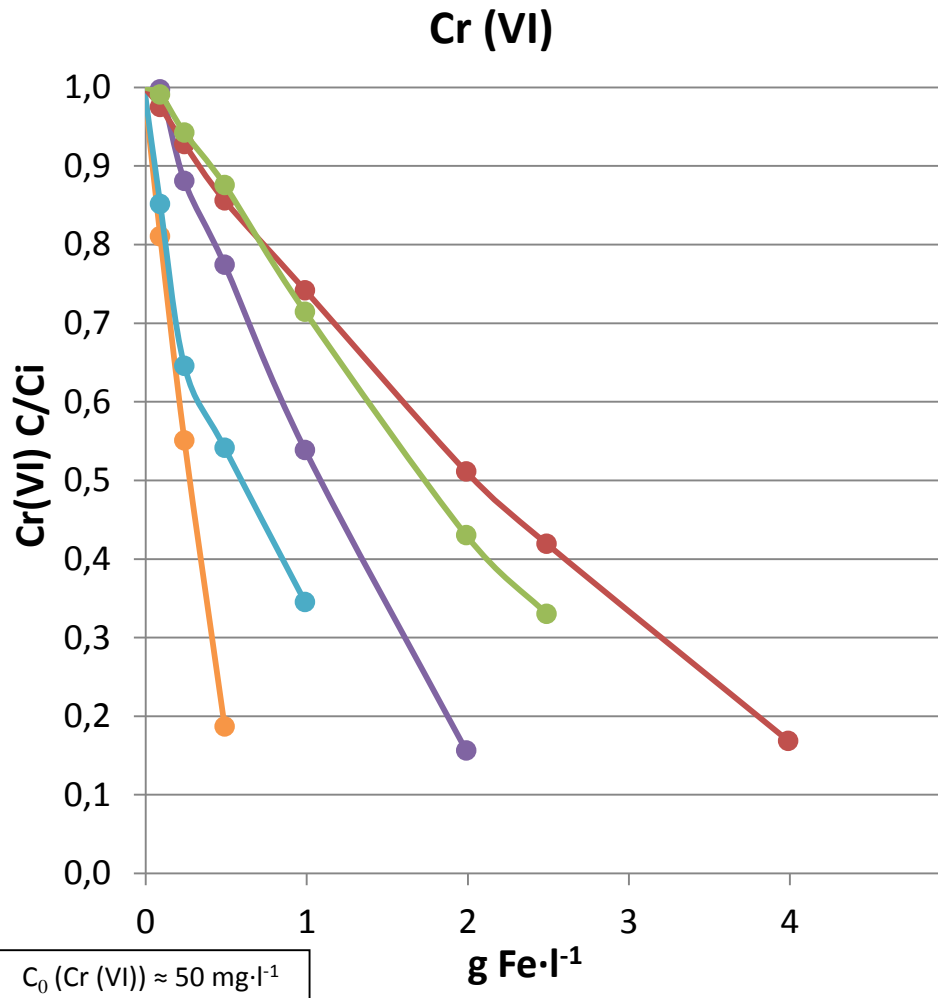
* Pyrophoric when isolated from MEG

¹ Köbert et al., 2014

Fe(0) content



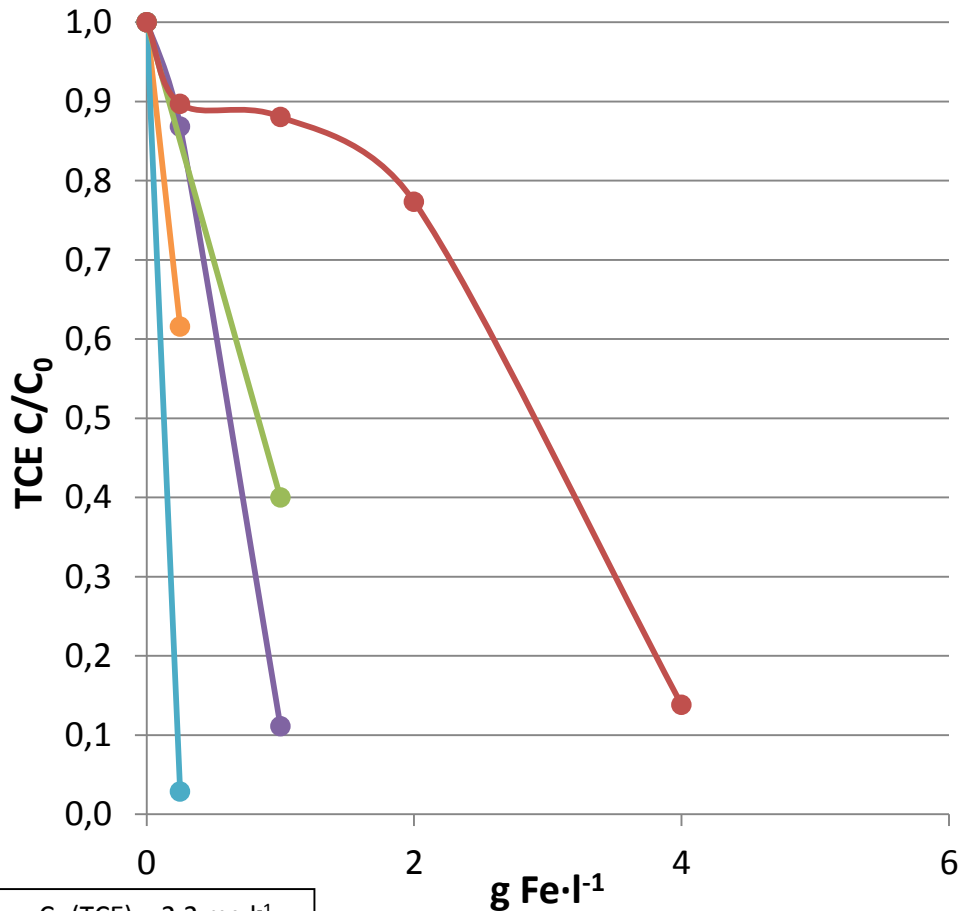
Reactivity Characterization: HM



| | Removal capacity | Removal capacity by SAA and Fe(0) |
|---------|-------------------------|---|
| | mg Cr(VI)·g Fe | mg Cr(VI)·g Fe ⁻¹ ·m ⁻² |
| NA 74 | -80.82 | -3,19 |
| NA 84 | -30.86 | -1.41 |
| 25P | -20.39 | -0.84 |
| STAR | -10.00 | -0.88 |
| UVR-FIA | -11.75 | -0.88 |

Reactivity Characterization: CAH

TCE



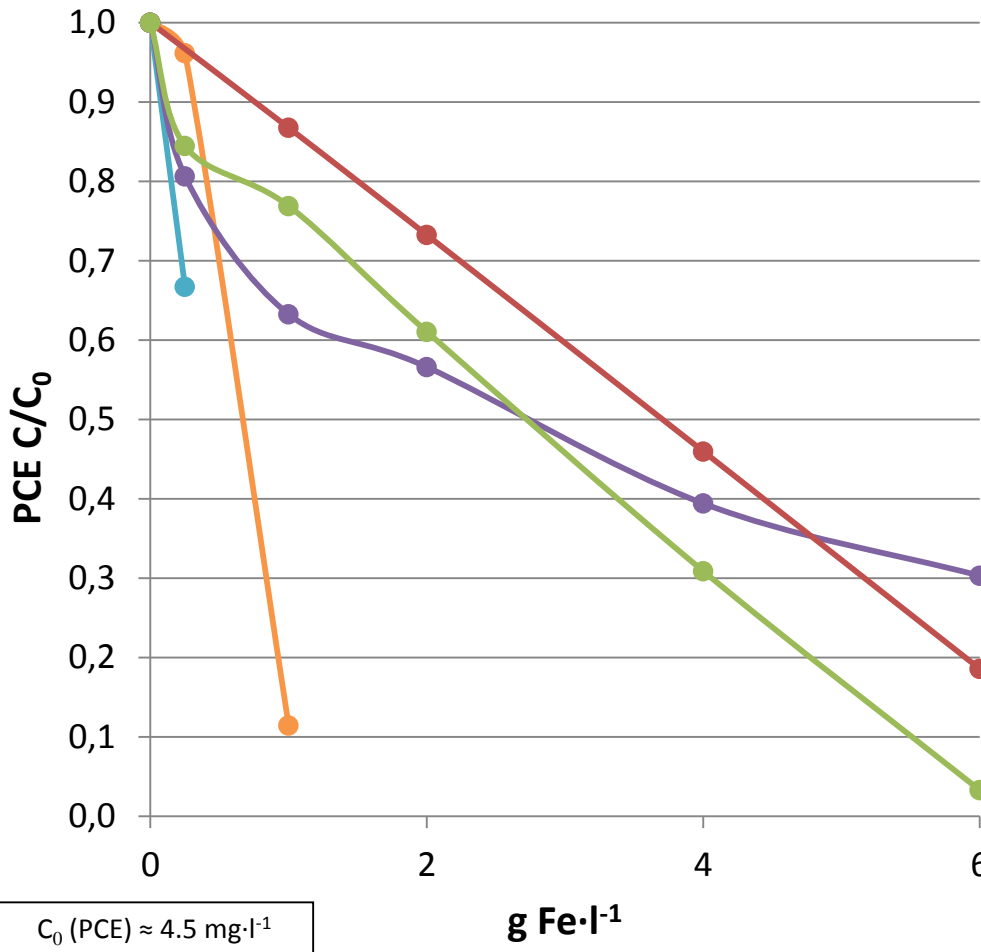
$C_0(\text{TCE}) \approx 2.2 \text{ mg}\cdot\text{l}^{-1}$

| | Removal capacity Mg TCE·g Fe | Removal capacity by SAA and Fe(0) mg TCE·g Fe ⁻¹ ·m ⁻² |
|---------|---------------------------------|--|
| NA 74 | -3.38 | -0.11 |
| NA 84 | -11.76 | -0.40 |
| 25P | -2.20 | -0.08 |
| STAR | -1.47 | -0.10 |
| UVR-FIA | -0.56 | -0.03 |

trans-1,2 DCE; cis-1,2 DCE; 1,1 DCE; VC
NOT DETECTED

Reactivity Characterization: CAH

PCE

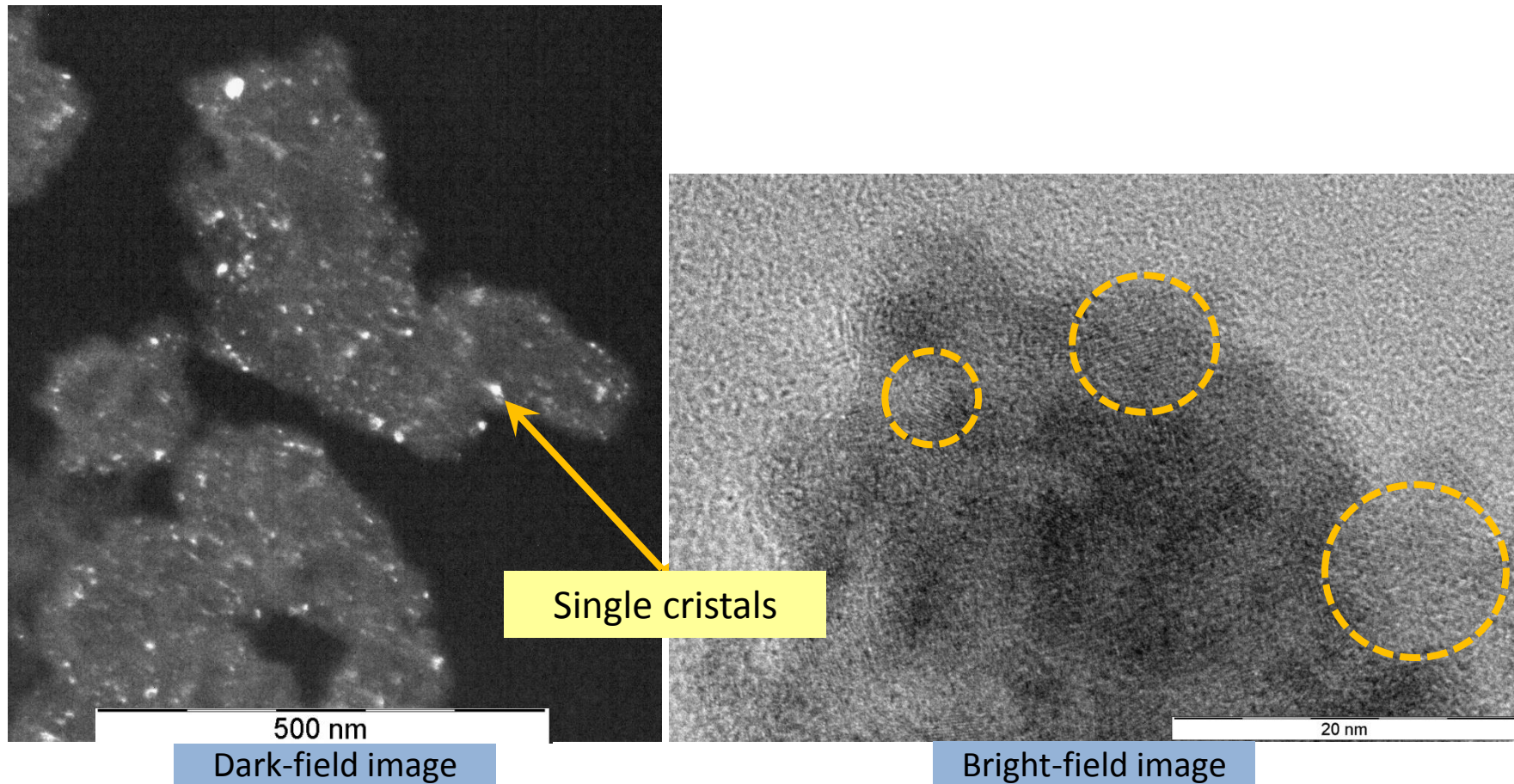


| | Removal capacity Mg PCE·g Fe | Removal capacity by SAA and Fe(0) mg PCE·g Fe ⁻¹ ·m ⁻² |
|---------|---------------------------------|--|
| NA 74 | -4.24 | -0.11 |
| NA 84 | -8.25 | -0.28 |
| 25P | -0.54 | -0.02 |
| STAR | -0.62 | -0.04 |
| UVR-FIA | -1.04 | -0.06 |

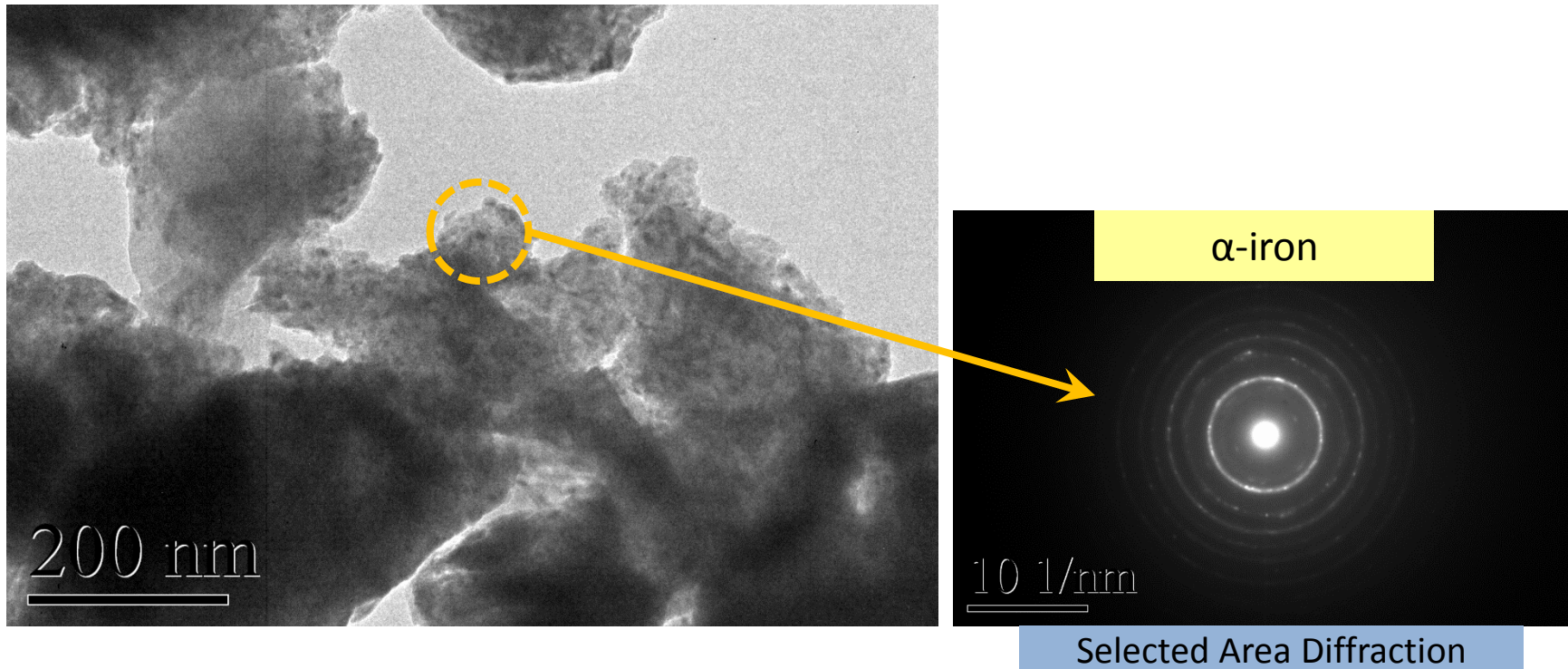
trans-1,2 DCE; cis-1,2 DCE; 1,1 DCE; VC
NOT DETECTED

C₀ (PCE) ≈ 4.5 mg·l⁻¹

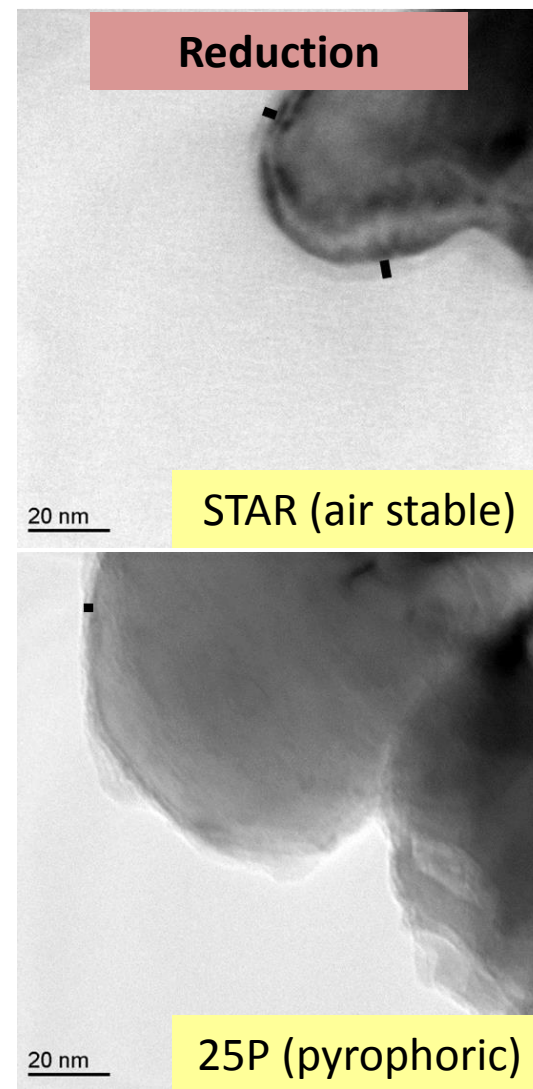
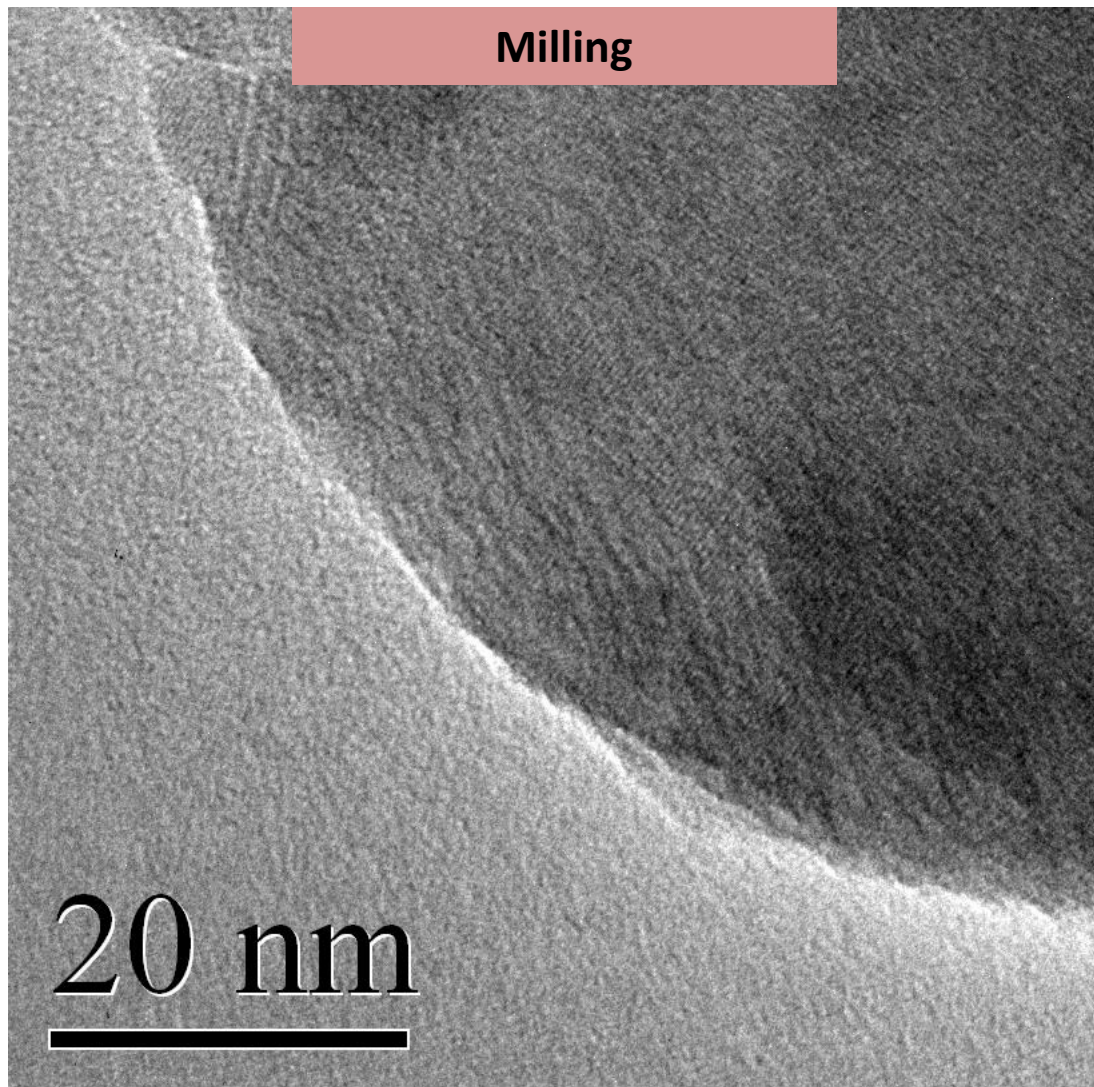
Reactivity and Structure: TEM



Reactivity and Structure: TEM



Reactivity and Structure: TEM



Conclusions

- ❖ New successful approach for nZVI production
- ❖ Optimal physical properties (Size, BET, %ZVI)
- ❖ Excellent reactive performance

Questions ???

