Explosives Coating via Advanced Cluster Energetics (ACE™) Fluid Energy Mill (FEM) Technology

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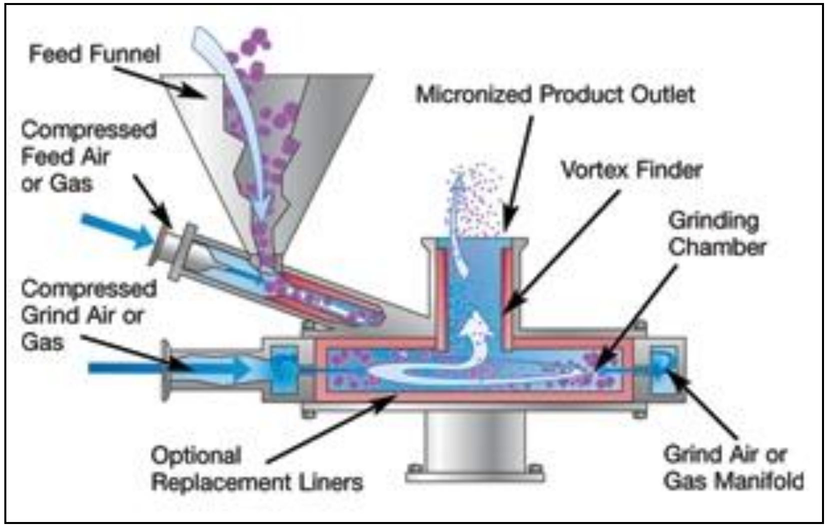
Background

- Advanced Cluster Energetics (ACE^{TM}) process developed jointly by New Jersey Institute of Technology (NJIT), Polymer Processing Institute (PPI) and RDECOM-ARDEC
- FEM technology utilizes compressed air to grind particles to less than 10 microns in size
- The ACE-FEM technology has potential to eliminate traditional coating processes
- Coated particles are subjected to particle to particle impact during the mill process where the coating is then re-distributed in-situ to the newly ground product
- Demand for smaller particle size materials to meet IM requirements is increasing

The ACE-FEM Process

- Pre-coated material added to mill system
- Feed air/grind air set to pre-determined position
- Feed rate adjusted for each material
- Product collected
- Analysis of product



Inert Trials

- 10 Milling Trials using inert materials
  - 5 Trials – 5% Wax; 95% Potassium Chloride (KCl)
  - 5 Trials – 3% Wax; 2% Dioctyladipate (DOA); 0.01% UV Tracer; 94.99% KCl

- Normally HSAAP utilizes slurry coating techniques to coat materials with wax or plastic
  - Due to solubility of KCl these techniques could not be employed
  - Drum coater used to coat KCl and Wax/DOA/Dye mixture
  - Prepared in 1 pound increments and blended for milling trials
  - Samples tested prior to milling
    - Two methods of analysis
    - Thermo Gravimetric Analysis (TGA)
    - Gravimetric Extraction

Wax/KCl Inert Trials (Input Coating)

- Input material prepared in 5 pound increments
- Drum coated and analyzed via TGA
  - Average = 5.992% Wax
  - Standard Deviation = 0.732
- Some variation from batch to batch but overall the wax did adhere to the KCl

Wax/KCl Input Batch	% Wax
Batch 1	6.053
Batch 2	5.799
Batch 3	5.297
Batch 4	5.668
Batch 5	7.784
Batch 6	5.502
Batch 7	5.740
Batch 8	6.552
Batch 9	5.397
Batch 10	6.126

Wax/KCl Inert Trials (Milling Trials)

- Milled in 10 Pound Increments
  - Feed Pressure = 80 PSI
  - Grind Pressure = 100 PSI
  - Feed Rate = 30 lb/hr
- Milling was uneventful resulting in free flowing powder
  - After 24 hours material did clump, most likely due to interaction between moisture and KCl
- Analyzed via TGA for wax content
  - Average = 5.359% Wax
  - Standard Deviation = 0.794
- Similar to input materials

Wax/KCl Milling Trial	% Wax
Trial 1	4.866
Trial 2	6.715
Trial 3	4.713
Trial 4	5.276
Trial 5	5.225

Wax/DOA/Dye/KCl Inert Trials (Input Coating)

- Input material prepared in 5 pound increments
- Drum coated and analyzed via gravimetric extraction with DMSO/Chloroform
  - Due to high volatility of DOA could not use TGA
  - Average = 2.16% DOA; 3.02% Wax
  - Standard Deviation = 0.18; 0.34
- Again variation from batch to batch but overall the KCl is coated with approximately 5% coating

Wax/DOA/Dye/KCl Input Batch	% DOA	% Wax
Batch 1	2.33	2.18
Batch 2	2.24	3.22
Batch 3	2.27	2.88
Batch 4	2.15	2.92
Batch 5	2.26	3.07
Batch 6	2.39	2.98
Batch 7	1.93	3.08
Batch 8	2.10	3.27
Batch 9	1.79	3.43
Batch 10	2.19	3.15

Wax/DOA/Dye/KCl Inert Trials (Milling Trials)

- Milled in 10 Pound Increments
  - Feed Pressure = 80 PSI
  - Grind Pressure = 100 PSI
  - Feed Rate = 10 lb/hr
- Material was not free flowing and continually compacted into the feed funnel
  - After 24 hours material again clumps, most likely due to interaction between moisture and KCl
- Analyzed via gravimetric extraction
  - Average = 2.16% DOA; 2.72% Wax
  - Standard Deviation = 0.03; 0.06
- Results in approximately 5% Coating

Wax/DOA/Dye/KCl Milling Trial	% DOA	% Wax
Trial 1	2.16	2.69
Trial 2	2.18	2.64
Trial 3	2.14	2.75
Trial 4	2.13	2.79
Trial 5	2.20	2.71

Live Trials

- 10 Milling Trials using energetic materials
  - 5 Trials – 5% Wax; 95% RDX
  - 5 Trials – 3% Wax; 2% DOA; 0.01% UV Tracer; 94.99% RDX

- Normally HSAAP utilizes slurry coating techniques to coat materials with wax or plastic
  - Due to use of drum coating in inert trials, it was decided to drum coat the live trials for consistency
  - Prepared in 1 pound increments and blended for milling trials
  - Samples tested prior to milling
    - Only one method of analysis
    - Gravimetric Extraction

Wax/RDX Live Trials (Input Coating)

- Input material prepared in 5 pound increments
- Drum coated and analyzed via extraction
  - Average = 4.42% Wax
  - Standard Deviation = 0.06
- Little variation from batch to batch

Wax/RDX Input Batch	% Wax
Batch 1	4.43
Batch 2	4.40
Batch 3	4.54
Batch 4	4.47
Batch 5	4.45
Batch 6	4.43
Batch 7	4.41
Batch 8	4.42
Batch 9	4.36
Batch 10	4.34

Wax/RDX Live Trials (Milling Trials)

- Milled in 5 Pound Increments
  - Feed Pressure = 80 PSI
  - Grind Pressure = 100 PSI
  - Feed Rate = 30 lb/hr
- Milling was problematic, multiple “blow back” events
  - Reduced feed rate to 20 lb/hr
- Material free flowing and unchanged after 24 hours in storage
- Analyzed via gravimetric extraction for wax content
  - Average = 4.73% Wax
  - Standard Deviation = 0.16
- Acceptable coating of RDX based on values

Wax/RDX Milling Trial	% Wax
Trial 1	4.52
Trial 2	4.63
Trial 3	4.73
Trial 4	4.89
Trial 5	4.89

Wax/DOA/Dye/RDX Live Trials (Input Coating)

- Input material prepared in 5 pound increments
- Drum coated and analyzed via gravimetric extraction with DMSO/Chloroform
  - Average = 2.27% DOA; 2.95% Wax
  - Standard Deviation = 0.05; 0.14
- Again variation from batch to batch but overall the RDX is coated with approximately 5% total coating

Wax/DOA/Dye/RDX Input Batch	% DOA	% Wax
Batch 1	2.20	3.00
Batch 2	2.28	3.18
Batch 3	2.28	3.06
Batch 4	2.24	2.85
Batch 5	2.23	2.91
Batch 6	2.23	2.99
Batch 7	2.36	2.99
Batch 8	2.30	2.64
Batch 9	2.35	2.95
Batch 10	2.26	2.93

Wax/DOA/Dye/RDX Live Trials (Milling Trials)

- Milled in 10 Pound Increments
  - Feed Pressure = 80 PSI
  - Grind Pressure = 100 PSI
  - Feed Rate = 10 lb/hr
- Based on milling of RDX/Wax the feed rate was reduced to 5 lb/hr
- Material continually compacted into the feed funnel similar to the inert Wax/DOA trials
  - However final product was soft and flowing even after 24 hours of storage
- Analyzed via gravimetric extraction
  - Average = 2.16% DOA; 2.72% Wax
  - Standard Deviation = 0.03; 0.06
- Acceptable coating of RDX based on values

Wax/DOA/Dye/RDX Milling Trial	% DOA	% Wax
Trial 1	1.98	2.89
Trial 2	1.99	2.91
Trial 3	1.98	2.92
Trial 4	1.98	2.88
Trial 5	1.99	2.99

Optimization Trials (Target Particle Size)

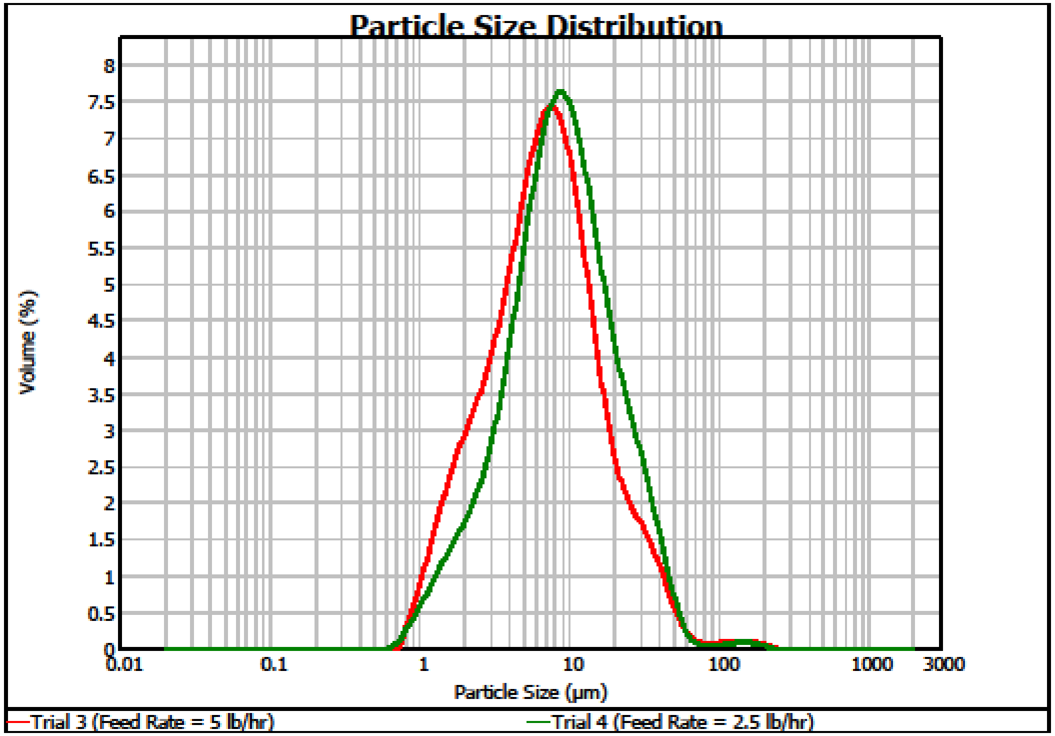
- Optimize grinding operations to match particle size of 4µm FEM RDX
  - Vary Feed Rate, Feed Pressure, and Grind Pressure using RDX coated with 5% DOA
  - Analysis for DOA content and particle size distribution after extraction

Sample ID	Feed Rate (lb/hr)	Feed Pressure (PSI)	Grind Pressure (PSI)	10th Percentile	50th Percentile	90th Percentile	DOA (%)
4µm FEM RDX				1.973	4.477	8.708	
Trial 1	5	100	100	5.235	19.172	59.164	4.66
Trial 2	5	110	100	1.911	5.956	16.654	4.95
Trial 3	5	110	110	2.027	6.869	21.139	4.92
Trail 4	2.5	110	110	2.709	8.702	25.632	4.88
Trial 5	5	100	80	2.045	6.231	17.193	4.83
Trial 6	5	110	110	2.345	7.268	20.769	4.74
Trial 7	5	100	80	2.398	5.730	14.110	4.69
Trial 8	5	100	75	2.859	7.590	18.931	4.64
Trial 9	5	100	75	2.693	7.408	18.143	4.80
Pilot Trial 1	5	100	80	3.052	8.346	20.079	4.71
Pilot Trial 2	5	100	80	2.814	8.935	27.274	5.03

Feed Rate (5lb/hr vs. 2.5lb/hr)

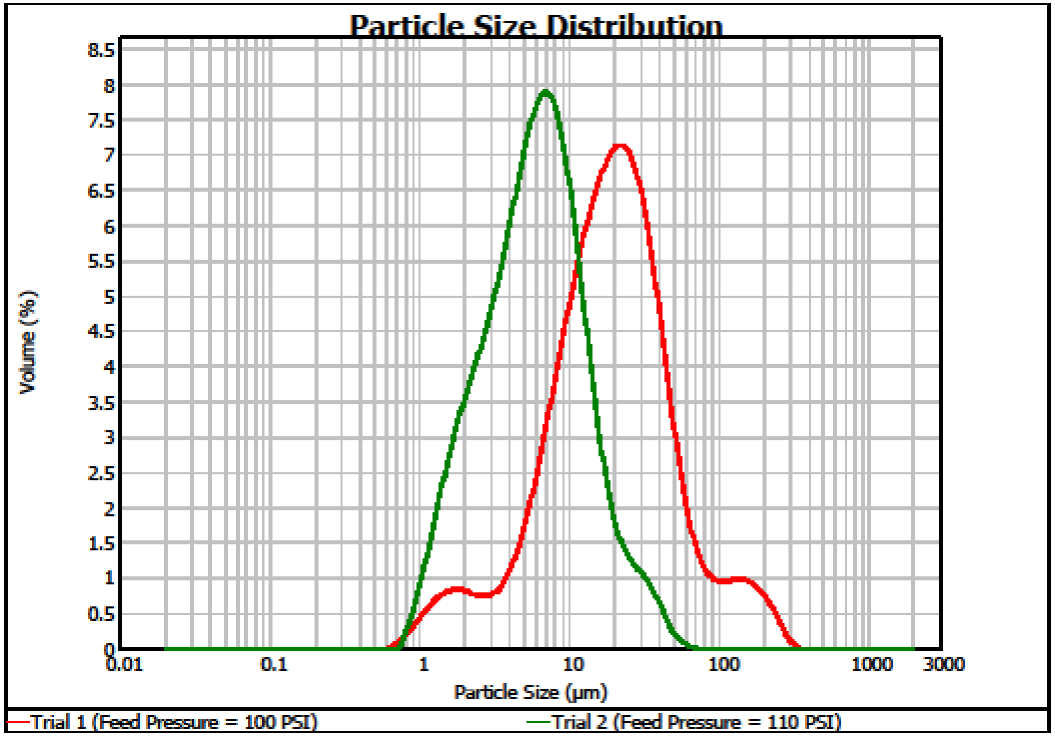
- Particle Size Analysis
  - Malvern Mastersizer 2000
  - Light Scattering Technique

- Changes made to feed rate
  - No real change in distribution
  - However neither sample matches 4µm FEM RDX target



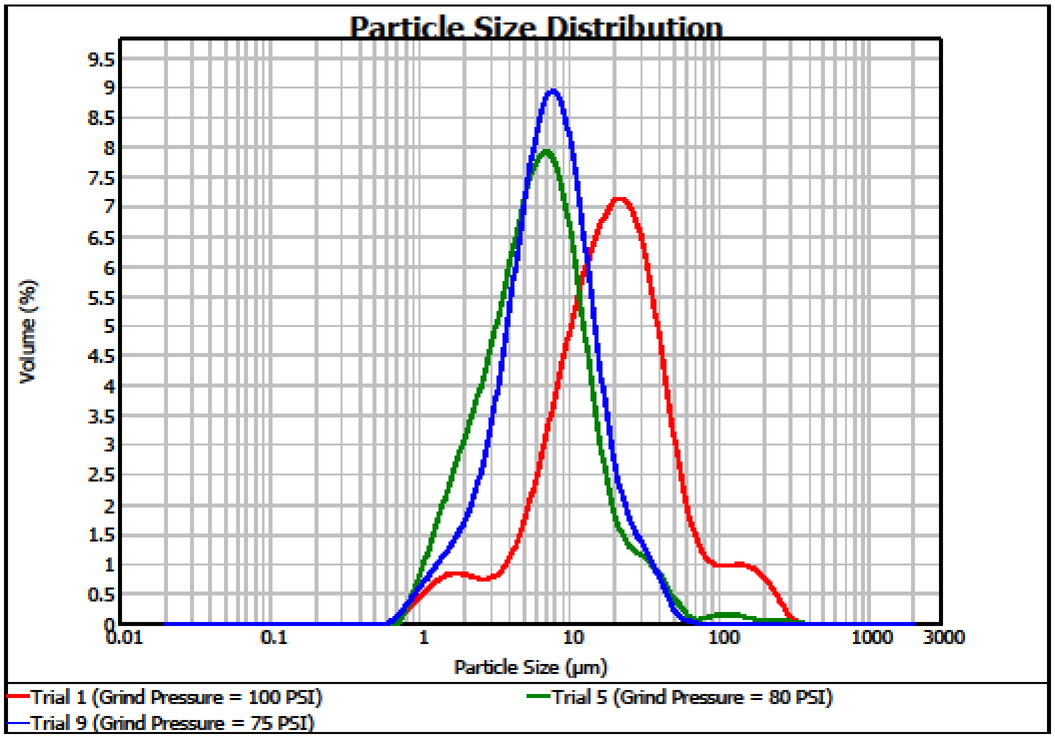
Feed Pressure (100 PSI vs. 110 PSI)

- Changes made to feed pressure
  - Lower pressure results in larger distribution
  - Neither matches 4µm FEM RDX



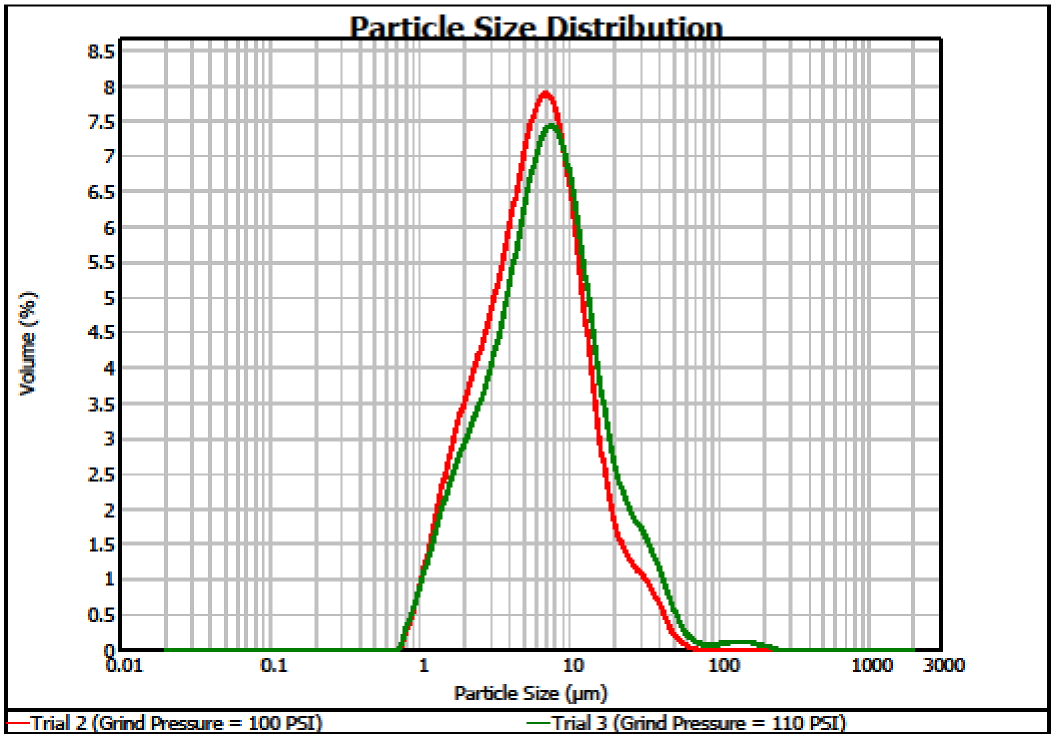
Grind Pressure at 100 PSI (100 PSI vs. 80 PSI vs. 75PSI)

- Changes made to grind pressure at constant 100 PSI Feed Pressure
  - Best distribution when compared to FEM RDX is at 80 PSI
  - Again no matches to 4µm FEM RDX



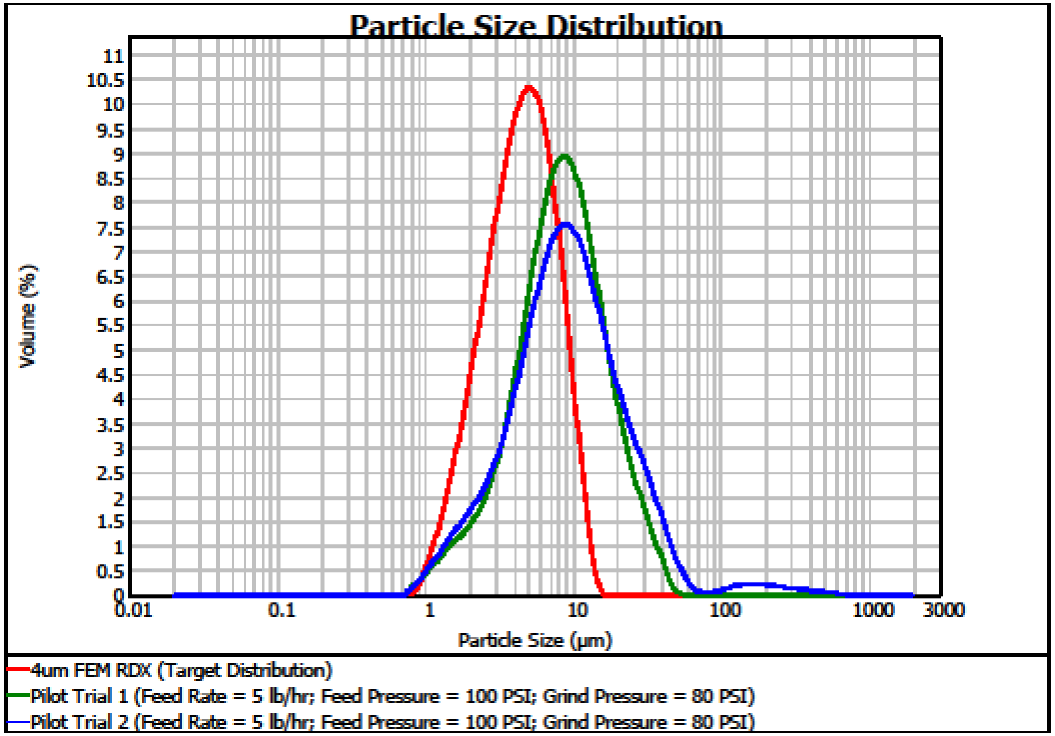
Grind Pressure at 110 PSI (100 PSI vs. 110PSI)

- Changes made to grind pressure at constant 110 PSI Feed Pressure
  - Very similar distributions to each other
  - No match to 4µm FEM RDX
- At this point the upper limit of the compressor was reached
  - In order to maintain higher pressures, a larger compressor is planned to be installed to continue evaluations



Evaluation of Process – Pilot Trials

- Decision to evaluate lower feed pressure
  - Feed Rate = 5 lb/hr
  - Feed Pressure = 100 PSI
  - Grind Pressure = 80 PSI
  - 5 pound batch size
- Particle Size Analysis
  - Run to run was excellent, only small variation in distribution
  - Still did not match 4µm FEM RDX target



Conclusions and Future Work

- ACE-FEM demonstrated the ability to reduce the particle size while re-distribution the coating
  - Inert trials result in similar coating content between milled material and input material
  - Live trials also result in similar coating values between milled and un-milled material, in some cases better coating with live material than inert
  - Some feed issues with DOA containing inputs
    - Changes in feed rates and or feeder type
  - Also did experience some “blow back” issues with live material
    - Possibly due to feeding
  - Optimization led to discovery that a different compressor was required to maintain higher pressure
- Future work on utilizing Scanning Electron Microscopy (SEM) to verify coating on surface with UV dye as well as different binder systems

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Questions?