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Surface Modified Color Pigments

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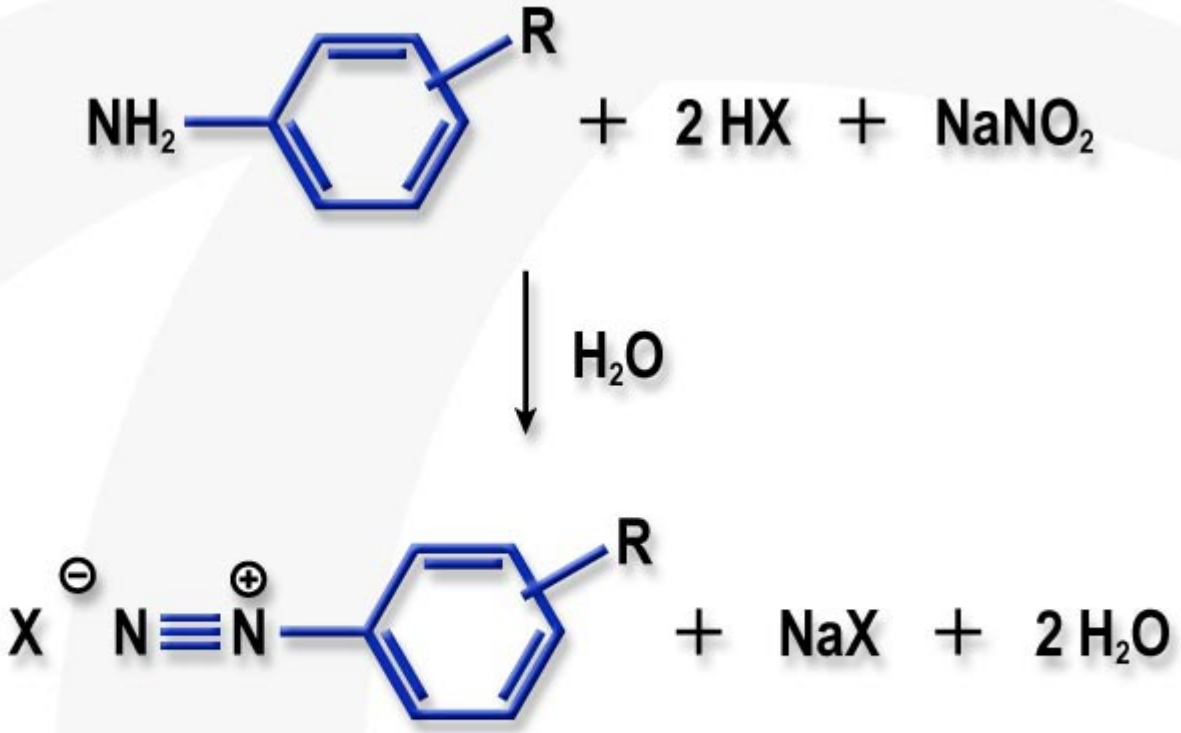
Outline

- Technology background
  - Diazonium Reaction
- Commercial products
- Extension to color pigments
  - Physical properties
  - Stability testing
  - Print properties
- Surface modification versatility
- Key benefits
- Summary

Diazonium Formation

US Patent 5,554,739
5,922,118



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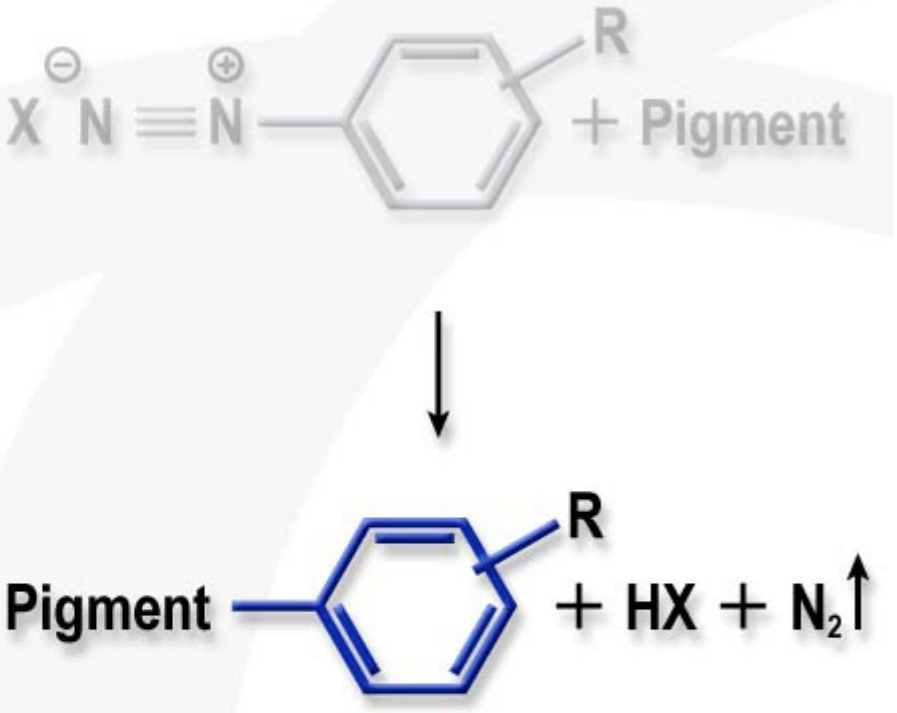
Surface Modification Reaction



R = COOH
= SO_3H
= Polymer

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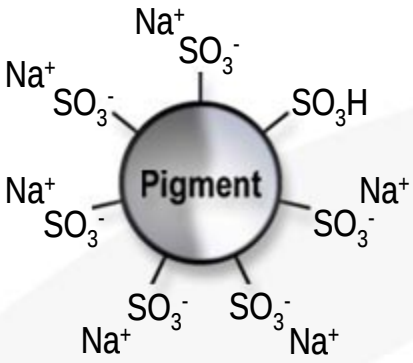
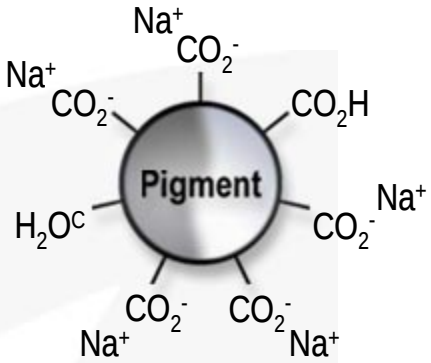
Example Commercial Products

Cab-O-Jet® 200

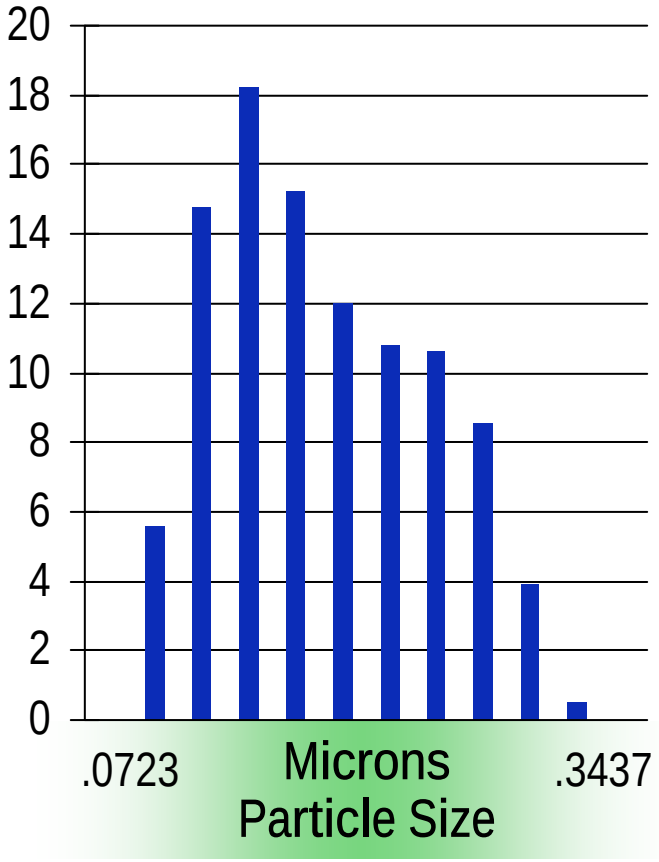


Cab-O-Jet® 300



Print Performance		
	COOH	SO_3H
Optical Density	1.5	1.4
Waterfastness	Yes	No

Frequency



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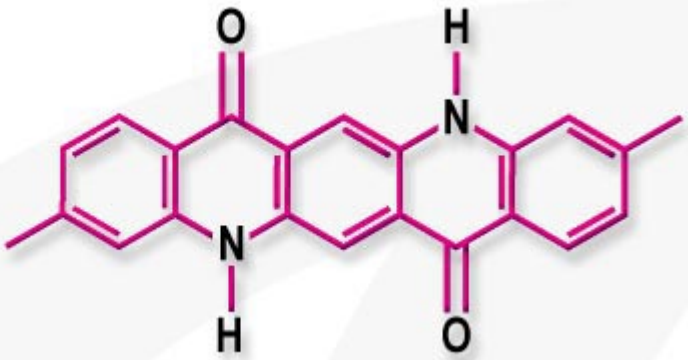
Extension to Color Pigments - Questions

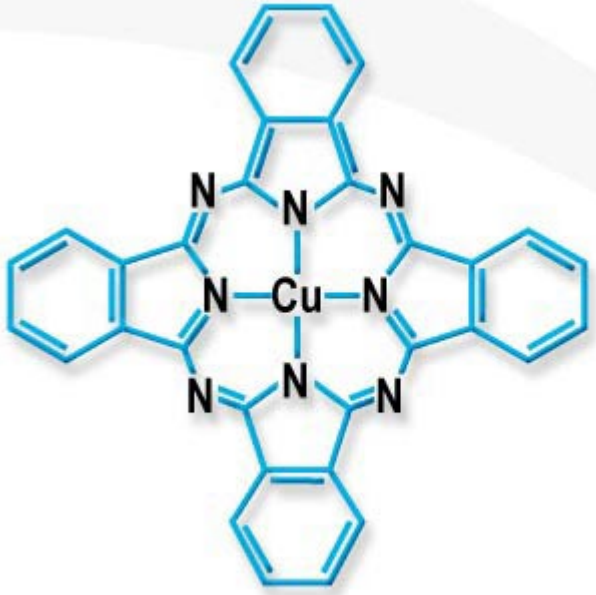
- Will diazonium chemistry work on organic color pigments
- By modifying surface with solubilizing groups will one create dye
- Will surface modification alter color properties and lightfastness
- Can we create a stable color pigment dispersion
- Will the approach be versatile

Extension to Color Pigments

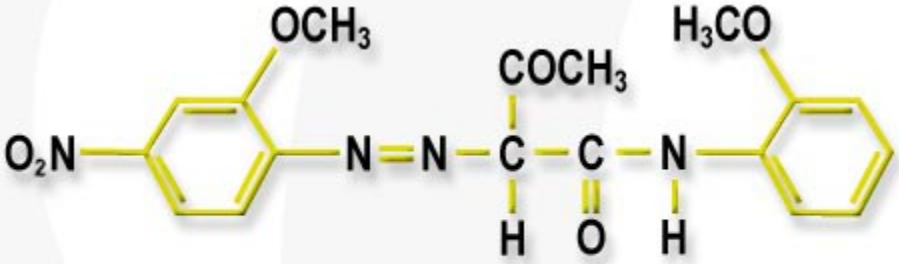
IJX266 — Quinacridone



IJX253 — Cu Phthalocyanine



IJX273 — Monoazo



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Physical Properties of Dispersions

Modified pigments with surface sodium sulfonate groups

Physical Properties	IJX™ 253C	IJX™ 266D	IJX™ 273B
Color	Cyan	Magenta	Yellow
Pigment Type	PB 15:4	PR 122	PY 74
Pigment Loading	11.7 %	10.4 %	9.2 %
Viscosity[1]	2.1 cP	2.4 cP	2 .0 cP
Surface Tension[2]	70.3 dynes/cm	71.8 dynes/cm	72.0 dynes/cm
pH	6.9	7.6	6.4
Particle size[3]	91 nm	105 nm	137 nm

[1]Shell #2 Efflux Cup
[2]Kruss Digital Tensiometer K-10
[3]Mean volume particle size determined by Microtrac® Ultrafine Particle Analyzer (Honeywell)

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Dispersion Purification

- Essential to remove reaction byproducts, excess salts and unreacted starting materials
- Purification is done using dialysis against DI water
  - Soluble impurities pass through membrane leaving surface modified pigment and its counterion
- Dialysis permeate does not contain solubilized dye molecules

Stability Testing

- Testing Conditions
  - Pigment dispersion at 10%
  - Generic ink with 5% pigment and 10% 2-pyrrolidone
  - Four-month aging 25°C and 70°C

Four Month Aging test of IJX™ 253 (PB 15:4)

PB 15:4	Mean volume particle size (nm)[1]		Number of particles > 0.5 µm[2]	
	INITIAL	AGED	INITIAL	AGED
25ºC: Pigment	92	92	2.7E+8	1.1E+8
Generic Ink	89	90	2.4E+8	1.6E+8
70ºC: Pigment	92	91	2.7E+8	1.8E+8
Generic Ink	89	90	2.4E+8	1.6E+9

[1]Mean volume particle size determined by Microtrac® Ultrafine Particle Analyzer (Honeywell)
[2]Determined by AccuSizer Model 780 available from Particle Sizing Systems NICOMP

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Four Month Aging test of IJX™ 266 (PR 122)

PR 122	Mean volume particle size (nm)[1]		Number of particles > 0.5 µm[2]	
	INITIAL	AGED	INITIAL	AGED
25ºC: Pigment	110	106	3.8E+8	1.4E+8
Generic Ink	105	106	4.0E+8	1.4E+8
70ºC: Pigment	110	94	3.8E+8	1.5E +8
Generic Ink	105	100	4.0E+8	1.3E+8

[1]Mean volume particle size determined by Microtrac® Ultrafine Particle Analyzer (Honeywell)
[2]Determined by AccuSizer Model 780 available from Particle Sizing Systems NICOMP

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Four Month Aging test of IJX™ 273 (PY 74)

PY 74		Mean volume particle size (nm)[1]		Number of particles > 0.5 µm[2]	
		INITIAL	AGED	INITIAL	AGED
25ºC:	Pigment	135	135	1.6E+8	5.8E+7
	Generic Ink	125	126	1.7E+8	1.3E+8
70ºC:	Pigment	135	130	1.6E+8	1.3E +8
	Generic Ink	125	105	1.7E+8	5.0E+7

[1]Mean volume particle size determined by Microtrac® Ultrafine Particle Analyzer (Honeywell)
[2]Determined by AccuSizer Model 780 available from Particle Sizing Systems NICOMP

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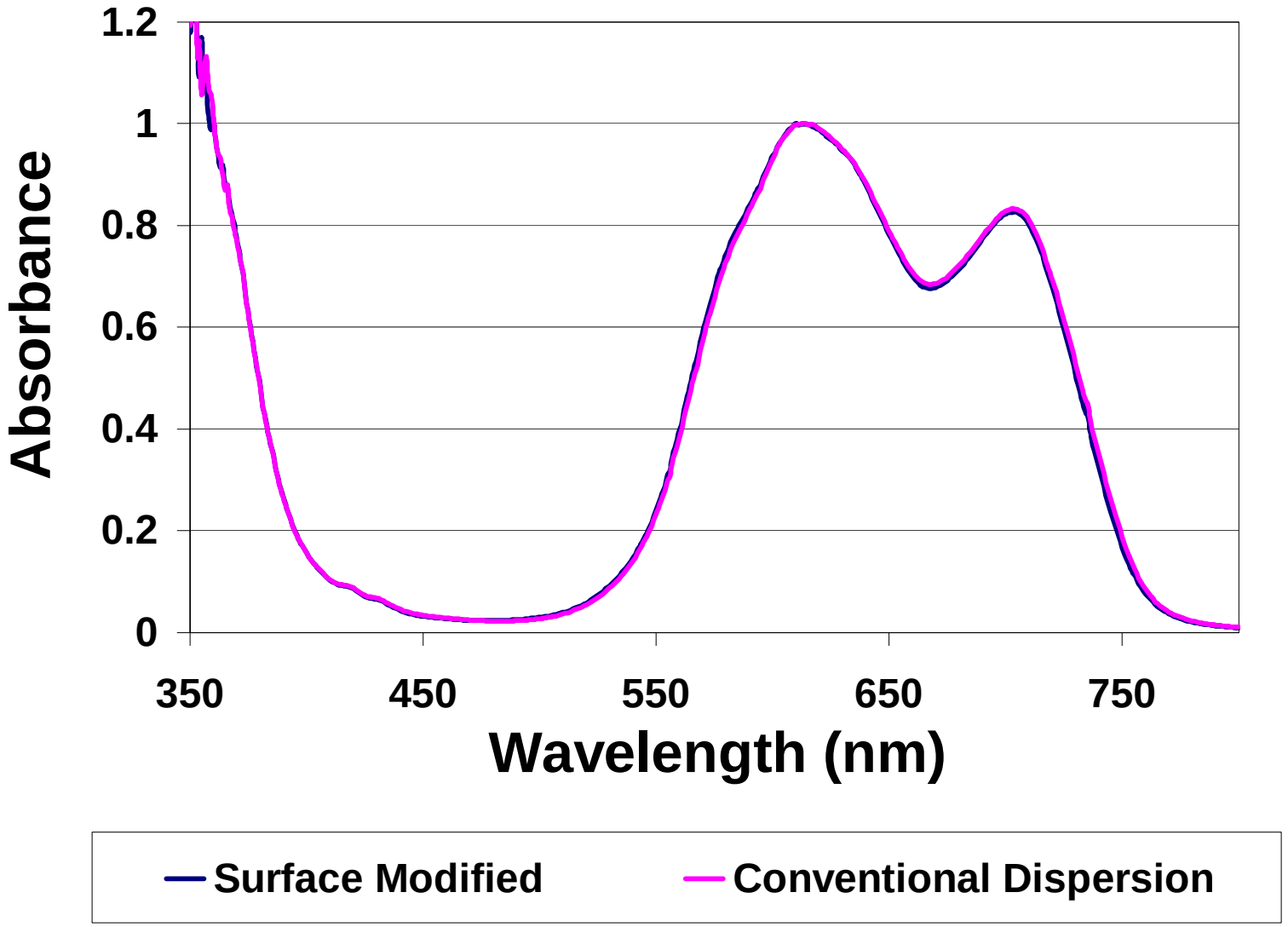
Impact of Surface Modification on Color

- UV-VIS absorbance
  - Comparison made at same pigment loading and same particle size
  - Compared surface modified to conventionally dispersed pigment
  - Compared nature of attached group - carboxylate versus sulfonate
- Result
  - Absorbance curves can be superimposed
  - No peak shift or additional absorbance seen

UV-VIS Comparison (PB15:4)



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Color Properties on Set of Plain Papers (PR122)

	Conventional	Surface Modified Pigments			
		Sulfonate		Carboxylate	Quat
		High	Low		
L*	57	55	56	53	54
a*	41	46	47	48	49
b*	-13	-12	-9	-13	-13
Chroma	43	47	48	50	51

Printing Performance

IJX™	Pigment	L*[1]	a*	b*	OD	WF[2]	LF[3]
253	PB 15:4	52	-18	-37	1.0	24 hrs	90%
266	PR 122	56	47	-9	1.0	5 min	93%
273	PY 74	89	-6	84	1.2	5 min	<50%

[1]L*a*b* readings determined by a Hunter LabScan II

[2]WF: Waterfastness is time taken by print to dry sufficiently that the runoff of 2.5 ml DI water does not cause colorant transfer

[3]LF : lightfastness expressed as % OD retention after 400 hrs of continuous UV-A irradiation using a Accelerated Weathering QUV/SE Instrument (Q-Panel Co.)

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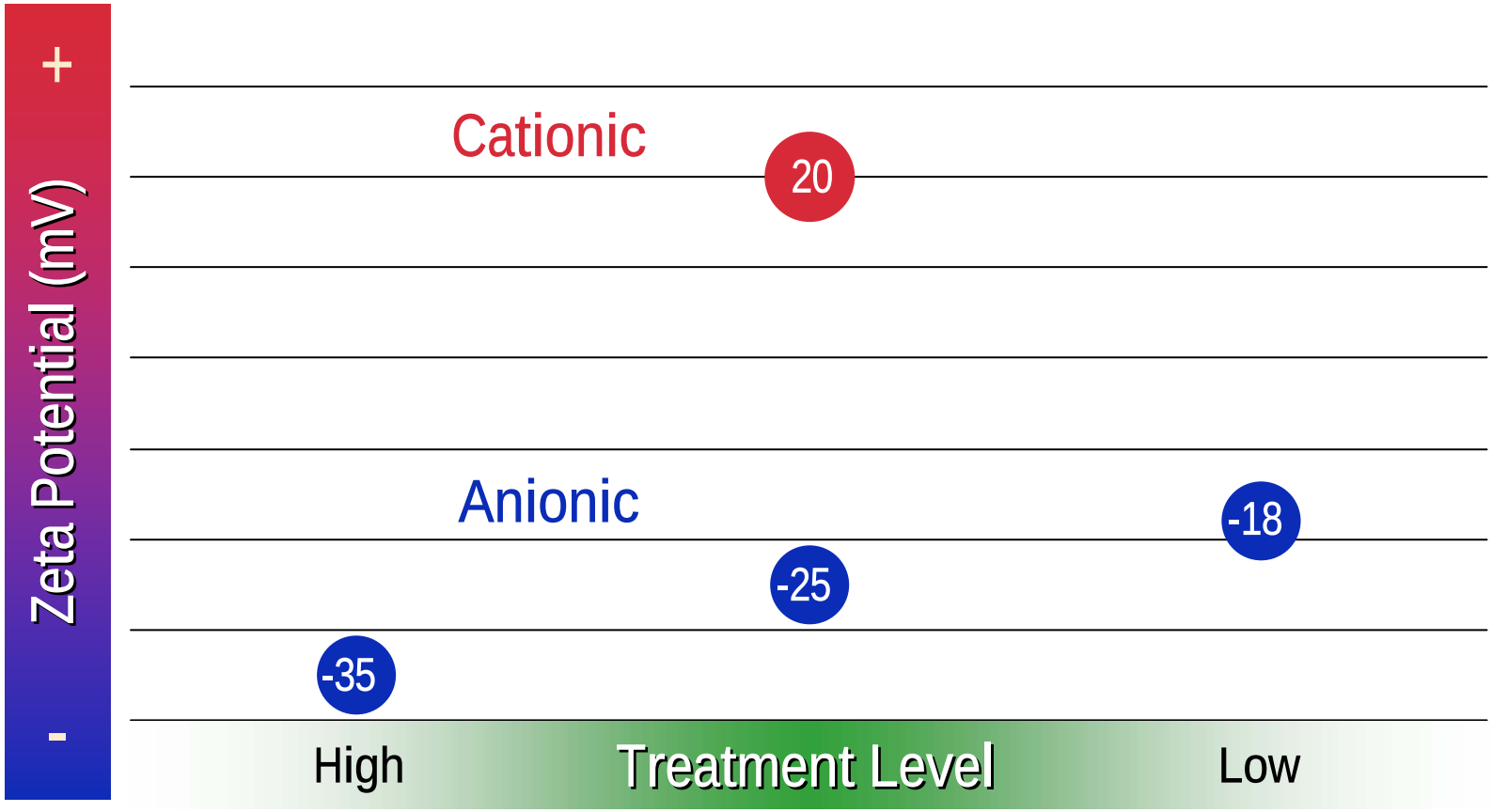
Surface Modified Color Pigments

- Conclusions
  - Particle size of all pigment dispersions and inks grew less than 10% after aging
  - Number of particles greater than 0.5 µm did not change after aging
  - Color and light stability of pigment seem unaffected by surface modification
  - No dye appears to be formed by surface modification

Zeta Potential



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Ion Exchange

Carboxylate sodium salt modified PB15:4

	Na^+ (ppm)	NH_4^+ (ppm)	Waterfastness
Before Ion Exchange	6000	—	1 hour
After NH_4^+ Exchange	90	4800	10 mins

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Benefits of Surface Modification

- Technology
  - Ability to tailor surface properties
- Physical Properties of Pigment Dispersions
  - High surface tension (~ 70 dynes/cm)
  - Low viscosity (< 2.5 cP at 10% solids)
  - Superior colloidal stability
  - High purity (material covalently attached)
- Ink
  - Formulation flexibility
  - No dispersants required
  - Superior reliability

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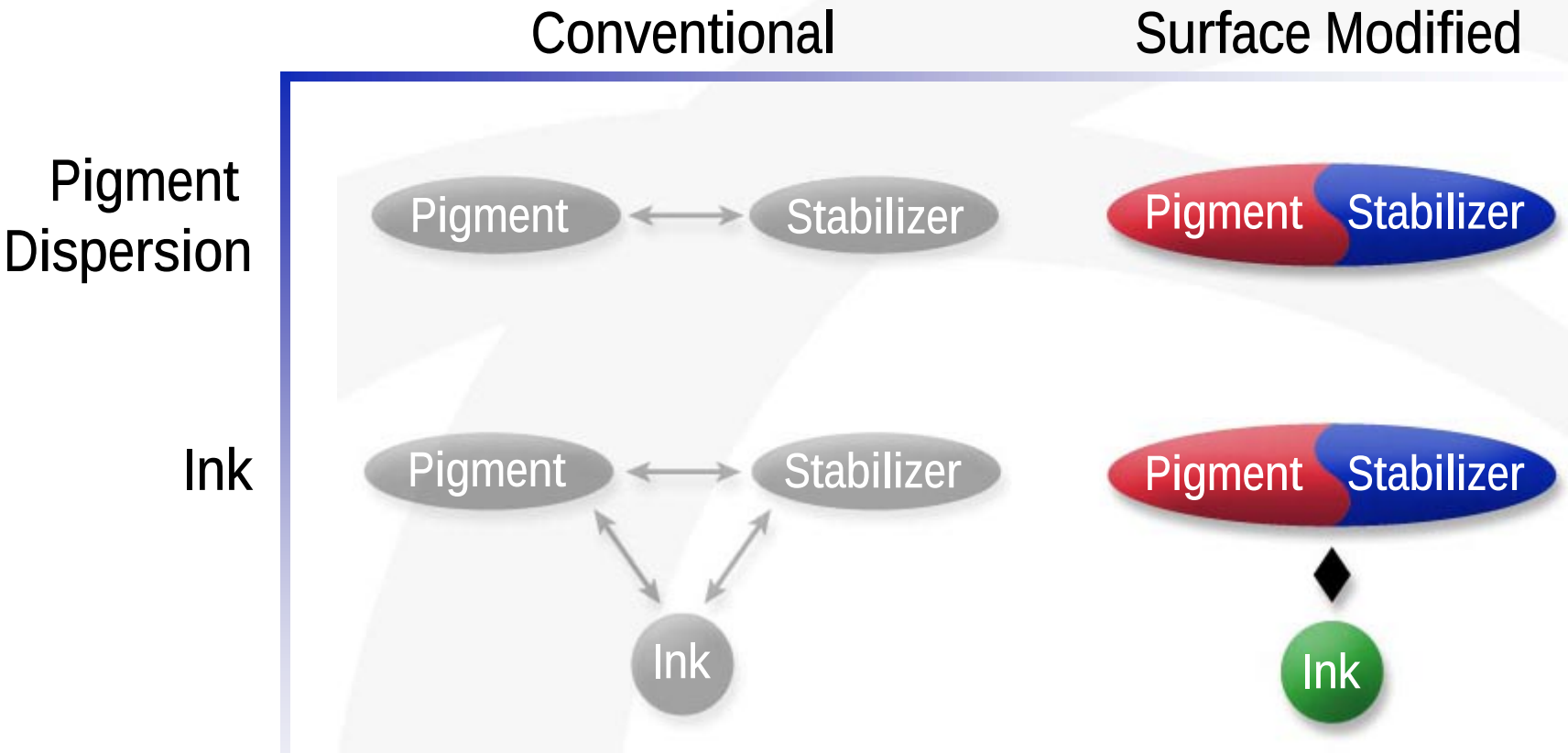
Formulation Flexibility



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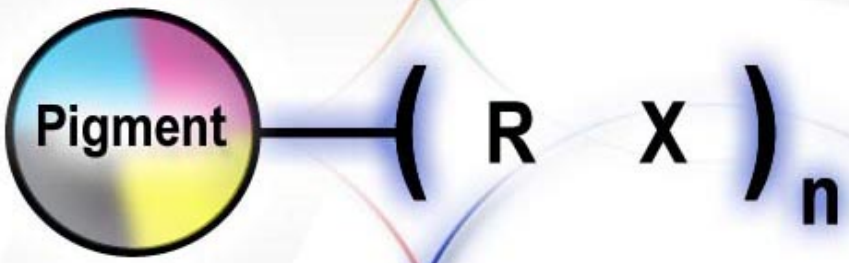
Surface Modification Versatility

Treatment Type

- Ionic (+ and -)
- Non-ionic
- Multiple/additional treatments
- Polymers

Counterion Type

- Negative/Positive
- Organic/inorganic
- Small molecules/polymers



Pigment Type

Black
Cyan
Magenta
Yellow

Treatment Level

adjusted for desired properties

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Broad Applicability to Pigment Classes

- Demonstrated surface modification of
  - Metal phthalocyanines
  - Quinacridone
  - Naphthol-AS
  - Mono-azo
  - Di-azo
  - Diketopyrrolo-pyrrole
  - Carbon Black
- Not all organic pigments can be treated

Summary

- Cabot surface modification technology extended to organic color pigments
  - Dispersing group attached to pigment surface
  - Provides unique and valuable dispersion properties
- Ideally suited for Digital Imaging Applications

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