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Coating Trial for an Antimicrobial Coating Containing Nisin Using a Large Scale Gravure Application Process

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Food Waste Problem

- Food spoilage - one of major causes of food waste
- Approximately 40% of food in the U.S. goes to waste
- Approximately 2/3 of products are lost due to spoilage in US households. (NRDC 2013)

Growth in Active Packaging

- Consumers demand fresh, minimally processed foods with same expectation of shelf life compared to foods with more processing
- Antimicrobial packaging – potential for shelf life extension via reduction of spoilage microorganisms
- Active packaging sector is expected to grow to 3.5 billion dollars by 2017 in the United States and 17.3 billion dollars worldwide. (Spinner 2014)

Objectives

- Formulate an antimicrobial coating suitable for large scale production methods
- Trial on large scale coating application processes such as gravure coater
- Verify that coated antimicrobial material inhibits spoilage microorganisms

Antimicrobial Nisin

- Non-toxic
  - Readily digested upon consumption
  - No odor or flavor
    - Mixtures containing salt may impart flavor compared to pure nisin
  - Slight coloration

- US FDA- GRAS approved (Generally Recognized As Safe)
- 10,000 IU/g (legal limit)
- 1 g pure nisin = 40,000,000 IU/g
- 2.5% nisin = 1,000,000 IU/g

Previous Antimicrobial Coating Work (Franklin et al 2004)

- Cellulose based coating
  - Methylcellulose/Hydroxyproply methylcellulose

- Coating was not heat sealable
- Low % solids (9.5%)
- Low pH (4.38)

- New formulation was produced from this work

Coating Formulation

Ingredient

- PVOH
- Vegetable glycerin
- Nisin
- Polysorbate 80

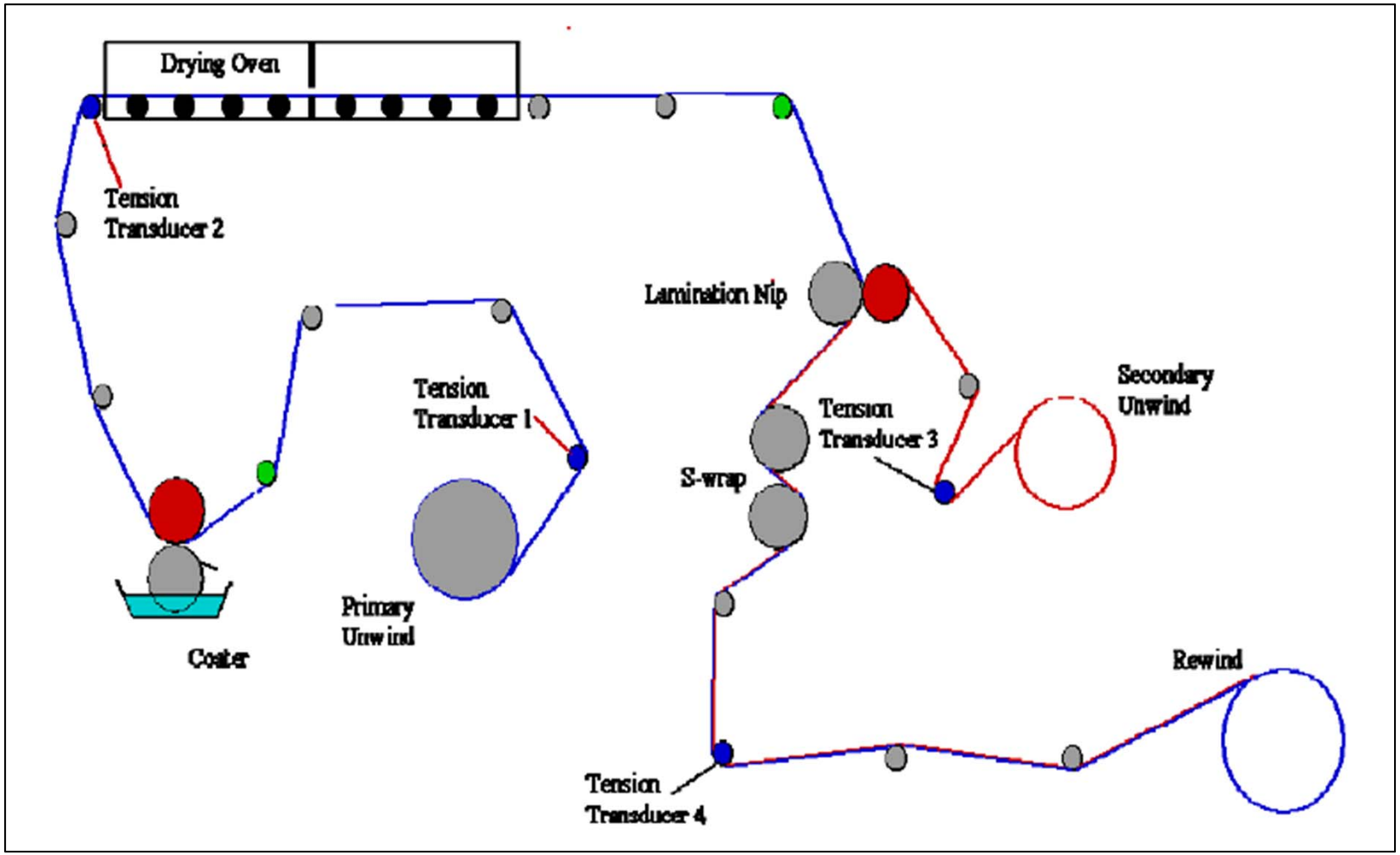
- Ethanol/water (50:50 v/v)
- 0.02 acetic acid solution

Purpose

- Thermoplastic (sealability)
- Plasticizer
- Antimicrobial component
- Surfactant, emulsifier, foam reducer
- Solvents
- Activate nisin, dissolution

Gravure Coater

Schematic



Rau 2009

Coating Formulation

Ingredient	Purpose
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• Nisin	• Antimicrobial component
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Control & Treatment Coatings

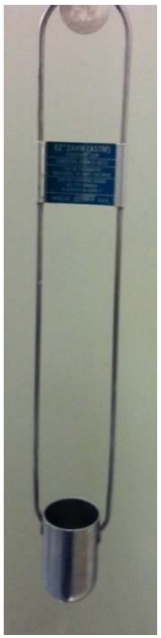
Substrate

- PET Coex
  - Typical structure for hot dog packaging
- Sealant layer: LLDPE
  - 32 dynes/cm

- Substrate was slit to 14.5” ww
- Corona treated (inline treater on OMET VaryFlex 530)
- Primed (PEI) (gravure coater in DuPont Laboratory in Newman Hall)

Selected Properties: Liquid Coating

- pH
  - pH meter – before trial
- Viscosity (Zahn #3)
  - Measurements before and after trial
- Percent solids
  - Measurements before and after trial

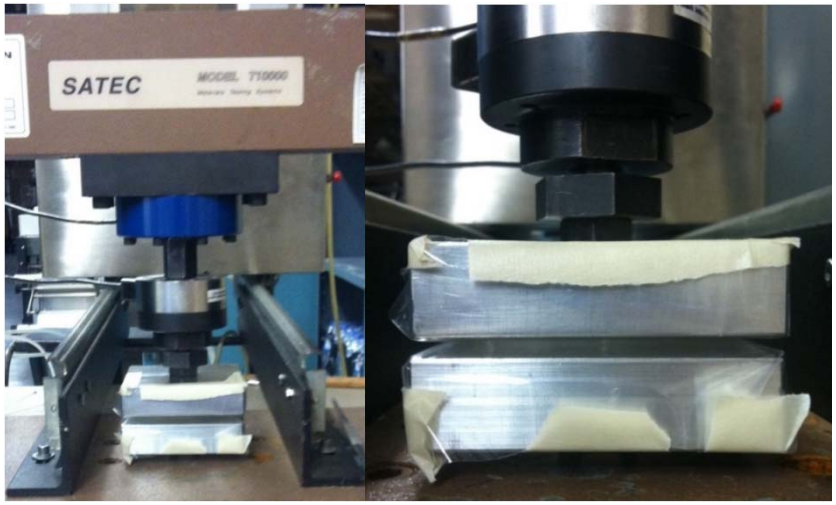


Conditions of Coater/Laminator in DuPont Lamination Laboratory			
Sample	Primer	Control	Treatment
Primary unwind material	48 ga PET/2.5 mil LLDPE Coex/ 48 ga PET		
Coat side	In	Out	Out
Tension (1° UW) (psi)	4	1.5	2.0
Web width (inches)	14.5	14.5	14.5
Rewind coat side	Out	Out	Out
Tension at rewind (psi)	10	10	10
Coater cylinder	200 Quad	110 Quad	110 Quad
Coating	Primer (PEI)	Control coating	Antimicrobial coating
Coating station tension (psi)	13	13	13
Dryer 1 temperature (°F)	155	155	160
Dryer 2 temperature (°F)	150	150	155
Line speed (ft/min)	26	25	25

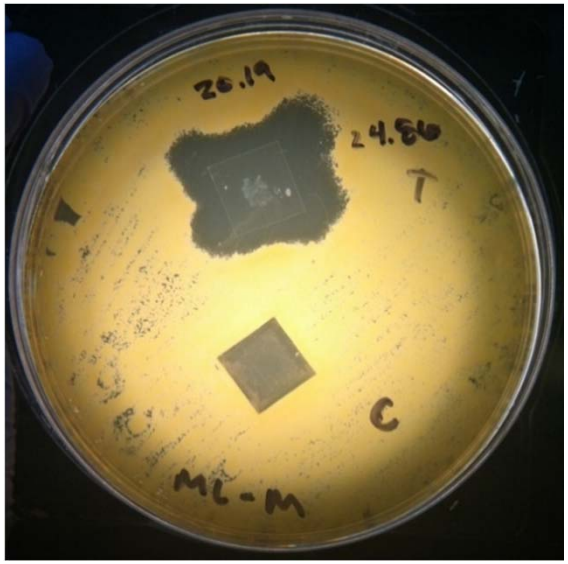
Material Produced

Selected Properties: Coated Material

- Coat weight
- Block testing



- Film on lawn
  - *Micrococcus luteus*
    - Spoilage indicator
- Haze



Gravure trial testing summary results for coatings and coated films			
	Control	**Antimicrobial**	**P values (α = 0.05)**
Solids content (%) (n = 60)	18.72±0.69	20.67±2.55	0.0002
Viscosity (sec) (n = 12)	BEFORE – 21.53±0.86 AFTER – 22.06±0.41	BEFORE – 20.10±0.72 AFTER – 17.67±0.12	<0.0001 C&T 0.0011 (treat* time)
pH (n = 11)	6.47±0.03	5.96±0.02	<0.0001
Basis Weight (#/ream) (n = 42)	1.50±0.13 (2.44±0.21 gsm)	1.48±0.20 (2.41±0.33 gsm)	0.7041
Block testing (gf) (n = 42)	290.60±94.86	321.35±52.89	0.2210
Haze (ΔE) (n = 40)	0.16±0.09	0.15±0.06	0.8675
Film on lawn (mm) *M. luteus* (n = 21)	0±0.0	5.78±2.20	<0.0001

Discussion of Selected Properties

- Slight increase in viscosity of coating after the trial due to solvent evaporation
- Solids content for control was 18.72% and treatment was 20.67%
- pH was slightly acidic (5.96 treatment; 6.47 control)
  - Corrosion resistant parts
  - Thorough cleaning

Selected Properties Continued

- Coat weight ~ 1.48-1.50 #/ream
  - 110 Quad cylinder
  - 25 ft/min line speed
- Blocking
  - Exceeded 200 gf limit listed in ASTM D3354
    - D3354-15: Standard Test Method for Blocking Load of Plastic Film by the Parallel Plate Method.
- Haze
  - Results were less than 1.0
  - $\Delta E < 1$ = imperceptible to the human eye

Selected Properties Continued

- Film on lawn
  - Effective against *M. luteus* (ATCC 10240)
  - Film samples 12.5 cm square
  - Treatment film produced 5.78±2.20 inhibition zone
  - Control film produced inhibition but no inhibition zone
    - Could be caused by other coating ingredients such as entrapped 0.02 M acetic acid

Film Quality: Troubleshooting

- Adhesive failure
  - Delamination of coating from substrate
  - WHY?

Troubleshooting

- Low coating adhesion
  - Drying issue?
  - Skinning
    - Retained solvents
- Primer
  - Improper application – coating dissolving primer
    - Contacted primer supplier
- Sealing
  - Back side treat
  - Excessive corona treatment
  - Coating components (WBL)
  - Drying issue -skinning

Troubleshooting: Retained Solvents

Sample	Ethanol Level (mg/ream)	Ethanol level (mg/m^2)	Ethanol Level (ppm)
Ambient dry 1	9	0.032	13.2
Ambient dry 2	12	0.043	17.6
Oven dry 1	24	0.086	35.3
Oven dry 2	21	0.075	30.9

Troubleshooting: Primer

- Contact with supplier
  - Used 200 LPI cylinder
  - Cylinder delivering too high wet weight…unable to fully dry
  - Recommended 400-600 LPI for no more than 0.5 #/ream laydown

Troubleshooting: Other

- Corona treatment – effect on LLDPE
  - Excessive corona treatment – 60 dynes
  - Crosslinking, decreased chain mobility, increased melt temp., fracturing film surface & group reorganization (Foster 2015, Farley & Meka 1994, Zhang, Sun & Wadsworth 1998)
- Plasticizer or Polysorbate 80
- Oily based component migrating to surface – WBL
  - Primary seal failure – peelable seal

Troubleshooting Conclusion

- Run a second trial
- OMET VaryFlex 530 Press in the Sonoco Institute of Packaging Design and Graphics
  - 400-600 LPI anilox for primer
  - Reduce corona treatment to desired 36-37 dynes

Conclusions

- Able to up-scale coating formulation to gravure trial
  - Adhesion difficulties
- Treatment coated material was effective against spoilage organism *M. luteus* compared to control $P<0.0001$

**Second trial conducted using previous recommendations

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THANK YOU FOR YOUR ATTENTION

QUESTIONS?

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