A NOVEL EXUDATION TEST METHOD

Jacyr Quadros Jr. Marcos Mendonça Roberto Piagentini Emerson Madaleno

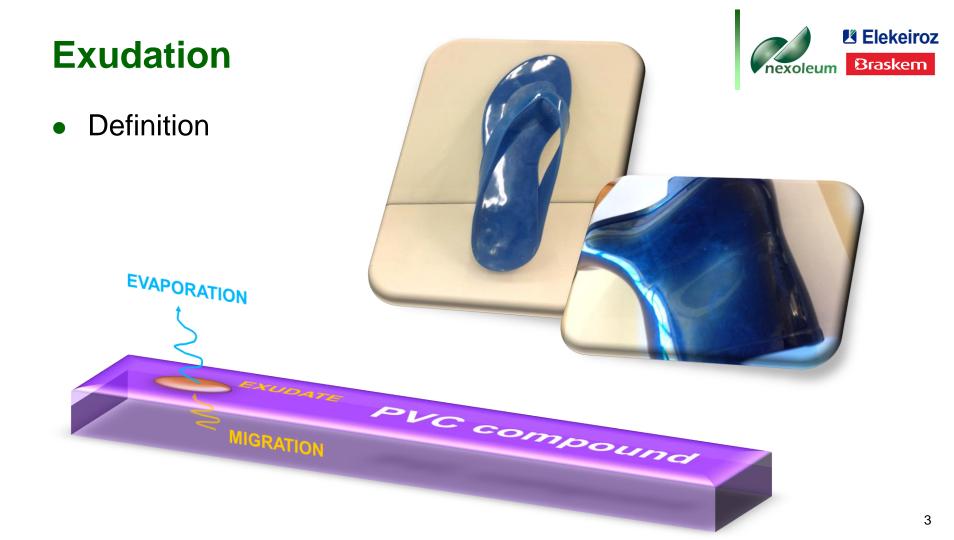


Braskem

Introduction



- Continued pursuit for sustainable solutions → new PVC plasticisers and additives
- Challenge: properly evaluate materials and compare properties
- Exudation
 - Highly relevant
 - Current existing tests: limited or in-house
- New exudation test method:
 - Quantitative
 - Comparative
 - Sensitive
 - Reliable



Basic principles

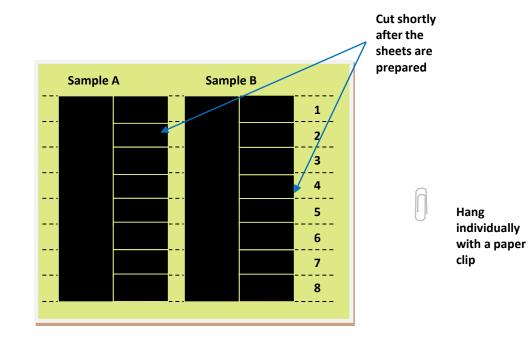
- Various measurements explored
 - Visual
 - Weight
 - Gloss
 - Surface Tension
- Three laboratories
 - All test specimens prepared at once
 - Different conditions
 - Different operators
- Various raw materials
 - Industry standards
 - Well known poor performers



Methodology Sample preparation



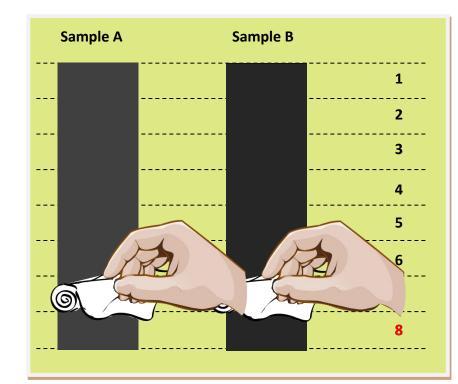
• Samples for weight and surface property measurements



Start: Nov/01

Procedures

• Preparation of "gradient" visual evaluation



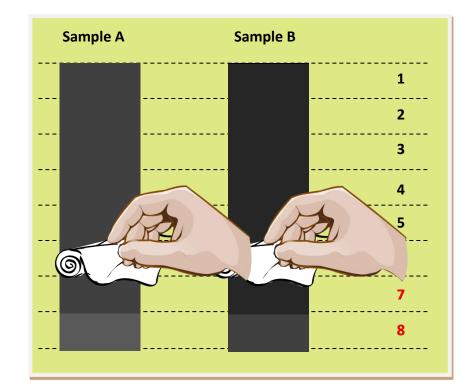


Nov/08

Start: Nov/01

Procedures

• Preparation of "gradient" visual evaluation





Nov/14

Nov/08

Procedures

nexoleum Braskem

• Preparation of "gradient" visual evaluation

A score from 0 (no visual exudation) to 5 (maximum visual exudation) was assigned to each "cell" at the end of the experiment,

Start: Nov/01

Sample A	Sample B		
0	0	1	Dec/26
0	0	2	Dec/19
0	0	3	Dec/12
1	0	4	Dec/05
2	0	5	Nov/28
3	1	6	Nov/21
4	2	7	Nov/14
5	3	8	Nov/07

8

Measurement procedures

- Mass measurements:
 - Each week, weigh corresponding test sample before and after cleaning
 - Mass variation is noted
 - Value is calculated in g/m² exudate
- Gloss and surface tension* measurements:
 - Each week, corresponding test sample is submitted to gloss meter and surface tension determination
 - Value is noted
- Visual evaluations:
 - After the end of 8 weeks, a score from 0 to 5 is given based on comparative evaluation of all test samples
 - Value is noted
- * Obs.: Surface tension measurements yielded no difference in measurement for all tested samples, therefore results were not considered in the discussion





Experimental design

nexoleum Braskem

- Sample size:
 - Strip width: 10 cm (split in two parts with 5 cm)
 - Strip length: 24 cm
 - Cell size: 3 x 4 cm
- Formulations:
 - Resin: 100 phr (type according to design)
 - Plasticiser (according to design)
 - CaZn Stabilizer: 1.5 phr
 - Black master batch: 7 phr

- Design (2^k):
 - For each plasticiser, 8 samples:
 - Thick and Thin sheets
 - Emulsion resin sample thickness: 0.25 and 0.50 mm
 - Suspension resin sample thickness: 0.50 and 1.00 mm
 - Low and High phr
 - Emulsion resin, plasticiser phr: 60 and 80
 - Suspension resin, plasticizer phr: 40 and 80
 - Emulsion and Suspension resins
 - Emulsion Resin: Norvin EP121 LM
 - Suspension Resin: Norvin P 1000



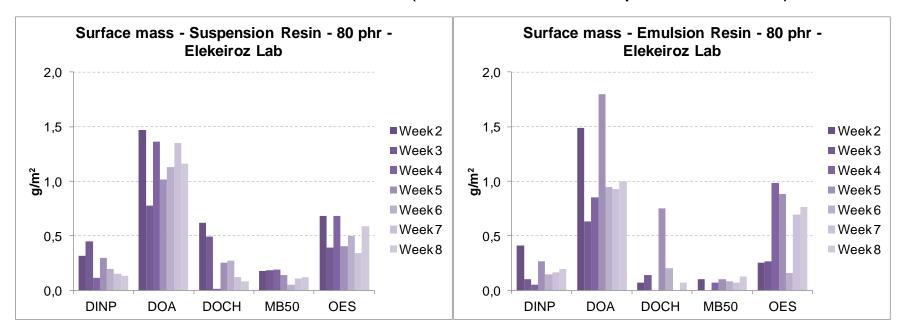
• Heavy exudation example





Results Exudation comparison

Surface mass measurement (emulsion and suspension resin)



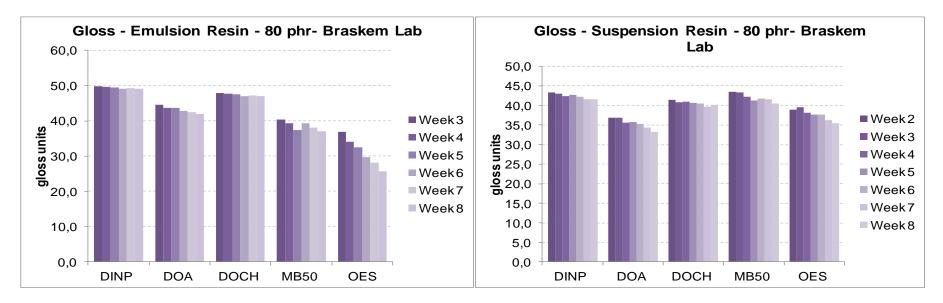
Obs.: Consistency of results for both types of resin



Exudation comparison



- Surface aloce measurement (emulsion and suc
- Surface gloss measurement (emulsion and suspension resin)



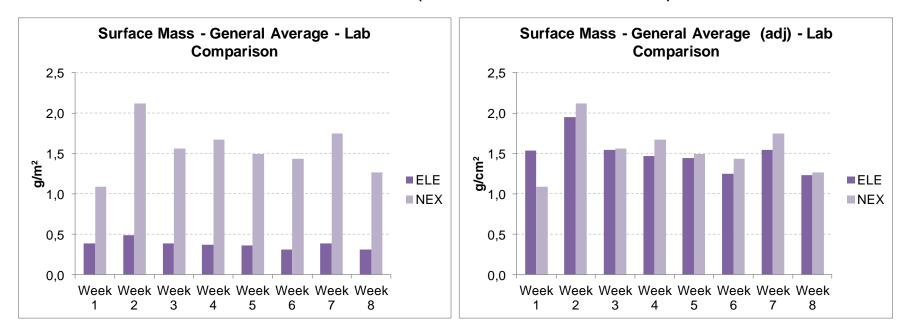
Obs.: Less consistency. Gloss is affected by "type" of exudation: hazy, greasy, waxy



Exudation comparison



• Surface mass measurement (different laboratories)

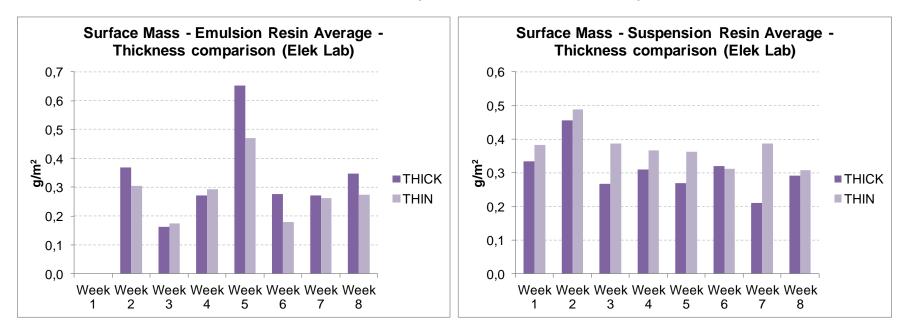


Obs.: Nexoleum lab ran under no temperature control. Elekeiroz @ 23°C, 50% humidity.

Exudation comparison



• Surface mass measurement (different thickness)

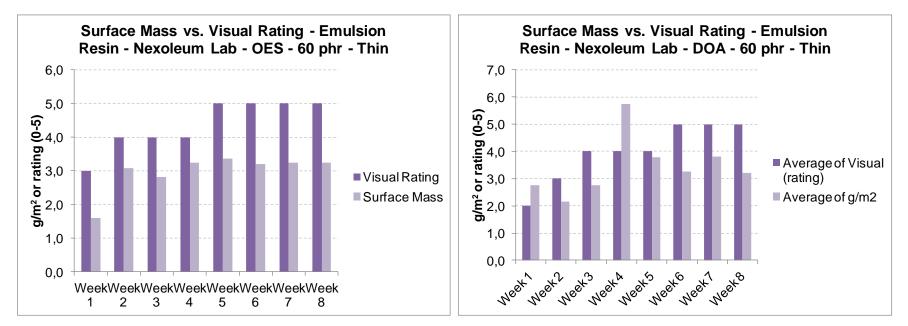


Obs.: Thickness appears to have very little effect on exudation

Exudation comparison



Surface mass measurement compared to visual rating



Obs.: Visual rating results fairly consistent with mass measurements

Conclusions



- Findings
 - Temperature and air movement greatly impact exudation
 - Thickness of the sample has little effect on exudation
 - Gloss and surface tension do not seem reliable as measurement tools
 - Visual observations are supplemental
 - Best and most consistent results obtained with surface mass measurements
 - Test has greater utility as a comparative tool
- Next steps
 - Initial results based on small sample, more data should be gathered to improve statistical significance
 - Interesting potential to be considered as a standard for exudation evaluations
 - Consistency of initial results
 - Simplicity of procedures
 - Key impact factors identified



THANK YOU