



On-line Monitoring of the Industrial Formaldehyde Resin Production



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Recommendation to reclassify formaldehyde emissions - summer of 2004

- Reduction of the resin mole ratio
- Need of reliable control of the whole resin synthesis procedure
- NIR spectroscopy has been applied to many fields of science incl. the wood based panel industry
- Spectroscopic applications developed and installed industrially by Chimar - GNOSSI™

Amino-resin industry

Raw materials → Final products

NIR technology & GNOSSI™

Synthesis procedure

Raw materials

UREA
FORMALDEHYDE

FORMOL UFC

Formaldehyde conc. Urea concentration

- Formaldehyde conc.
- Molar ratio
- Solids content
- Buffer capacity

UFC analysis of storage tanks

UFC Solids (molar ratio)

UFC MR (molar ratio)

NIR algorithms for UFC analysis

GNOSSI™ offers reliable, rapid, precise, non-destructive, on line, real time measurements

GNOSSI™ UFC

Based on tank analysis, the absorber is adjusted and continuously monitored.

UFC storage tank

compositions
molar ratio
solids
buffer capacity

GNOSSI™ UFC probe installation

Industrial results - GNOSSI™ UFC

% UREA	% FORMAL	% SOLIDS	MR
24.77	57.14	81.41	4.71

GNOSSI™ for UFC analysis

- REAL TIME PRODUCTION CONTROL
- ONLINE DETERMINATION OF SOLIDS AND MOLAR RATIO
- IMMEDIATE TIME OF RESPONSE
- NO MANPOWER REQUIRED FOR ANALYSIS
- NO MAINTENANCE
- LOW CONSUMABLES

GNOSSI™ for resin's synthesis

...the time to look through materials to confirm and prove what is theoretical believed just arrived...

Chimar Hellas - GNOSSI™ software

...the time to install an eye in the reactor to confirm its content and the progress of the condensation just arrived...

U & F Methylation & Condensation reactions

GNOSSI™

Molar ratio
Solids content
Progress of reactions

GNOSSI™ software is the unique technique that can simultaneously determine numerous parameters of a complex system. When installing a probe in the reactor it stops being a black box.

Industrial application of GNOSSI™

NIR tank probe
NIR probe installed

- Practical installation system that allows the daily cleaning of the probe.
- Depiction of the results on an excel file

Predicting MR & Solids

MR_UV	MR_UV	MR_UV	MR_UV
4.97	58.55	4.97	58.55

Calculated values: MR = 4.91 & Solids = 58.55

Monitoring the methylation stage

Methylation was completed in 5 min instead of expected 10 min.

Monitoring the condensation stage

The condensation reaction should follow the "standard pathway".

Industrial example of GNOSSI™ in resins

pH was corrected at 4.3 instead of 5.3

Predicting the end of the condensation

New path tends to

The prediction of the end of the condensation is impossible to be based only on the index.

Spectroscopic correlation of the progress of the viscosity during the reaction

Good correlation between NIR and Brookfield results allows predicting when to cut the reaction.

Industrial example I

The progress of the viscosity is predicted by GNOSSI™ with excellent precision.

Industrial example II

At the end of the condensation the progress of the viscosity is very rapid. Without GNOSSI™ it would be impossible to measure with Brookfield viscometer

Spectroscopic correlation of the molecular weight during the condensation stage

GPC analysis of samples during the condensation stage

Industrial example I

Mw and Viscosity during condensation of a UF resin (rapid reaction)

Industrial example II

Mw and Viscosity during condensation of a UF resin (rapid reaction)

We afford three spectroscopic models that follow the progress of the condensation:

- the condensation index,
- the progress of the viscosity and
- the progress of the molecular weight.

When combining all these we can predict the end of the condensation.

But... have we reached the goal?

...100 experiments that fulfill a law aren't enough for proving that a theory is correct. One experiment that doesn't fulfill a law is adequate for proving that the theory is wrong...

The area between the curve & a line parallel to axis x that passes from the maximum value of the index is constant.

Determination of the final molar ratio

MR_UV	MR_UV	MR_UV	MR_UV
1.074	74.31	1.074	74.31

Expected values

MR_UV = 1.070

Solids_UV = 74.23 %

The unique obstacle is

that GNOSSI™ software should be adjusted to the settings of each resin plant

CONCLUSIONS I

- Monitoring the production of raw materials (formol and UFC)
- Determine the quality of the stored raw materials allowing the adjustment of the absorber for keeping the production in specs
- Determine the molar ratio of the mixture each time a new component is loaded in the reactor
- Monitoring the progress of the methylation stage
- Monitoring the progress of the condensation stage in terms of viscosity and molecular weight of the polymer

CONCLUSIONS II

- Detecting abnormalities during the condensation stage
- Determine the properties of the final resin either in the reactor or in the storage tank
- As a technique it is non destructive precise reliable reproducible at real time & in situ