Why some migration conditions for plastics are not appropriate for other FCMs.

Peter Oldring
Representing numerous associations directly or indirectly involved in FCMs
FPF October 2015Zurich

DISCLAIMER

- I am not an analyst.
- I cannot do analytical chemistry.
- However, I have to understand and work with – make decisions etc., – analytical data, in order to determine the safety of my company's products.
- The work is embryonic and associations are still joining and the final format will certainly be different to that initially envisaged, but as of yet we do not know how different.

Associations & Bodies participating in Initiative

ACE	Beverage cartons
APEAL	Steel for packaging
CEFIC-FCA	Suppliers of food contact additives
CEPE	Can coating manufacturers
CEPI	Paper industry
EAA	Aluminium
EEA	Porcelain enamelled articles
EMPAC	Rigid metal packaging
ETRMA	Rubbers
EuPIA	Printing inks
EWF	Wax federation
FEC	Housewares, all materials, non-stick for this work
FEFCO	Corrugated packaging
FEICA	Adhesives and sealants
FPE	Multi-material flexible packaging
GAE	Glass Alliance Europe
CELIEGE	Cork
CES Silicones Europe	Silicone elastomers
INSTITUTE NEHRING	Test house
JRC – as observers	

The Issue - 1

- In the Plastics Regulation (10/2011) and supporting documentation, i.e. Migration Guidelines, some of the simulants, times and temperatures are inappropriate for non-harmonised FCMs (Food Contact Materials).
- However in the absence of harmonised regulations the conditions used in the Plastics Regulation are often applied to non-plastics.
- National Regulations for non-harmonised FCMs are tending to adopt the conditions in 10/2011 – example proposed coating regulations for both Netherlands and Belgium, although different in detail.

The Issue - 2

- Obtaining higher levels of migrants under inappropriate conditions may result in mis-interpreting the results, particularly if the simulant, time and temperatures specified in 10/2011 cause deterioration or physico-chemical change of the substrate leading to an overestimation of migration compared to that in foodstuffs.

Industry response - 1

- Members of associations have formed a task to offer testing better adapted to the specificity of various materials/ sectors.
- JRC was requested to have an advisory capacity as they were responsible for leading the drafting of the technical guidelines for plastics (reg. 10/2011) and have currently a brief to look at non harmonised FCM
- Each sector is assessing the applicability or not of the plastics 10/2011 migration testing guidelines for their own sector.

Industry response - 2

- In some cases they are applicable for other FCM but in some cases they are not
- Gaps or lack of feasibility of implementations are identified and expertise collected and shared to offer technical solutions for improved compliance testing.
- Test proposals are based on technical / scientifically demonstrated justification.
- The Task Force are developing their own compliance guidelines with separate chapters for each non-harmonized FCM:

Industry response - 3

- Chapters
  - Introduction
  - FCM specific chapters
    - FPE
    - Adhesives
    - Light metal packaging
    - Paper & Board
    - Silicones
    - Rubbers & TPE
    - Coatings not covered elsewhere
      - Further sub-divided: work in progress

Industry response - 4

- Can – EMPAC / CEPE
- Heatseal – EuPIA / FEICA / FPE
- Coldseal - EuPIA / FEICA / FPE
- Coatings on plastic film CEPE (part)
- Non-stick – FEC / CEPE / CEFIC
- Coatings on Paper and board – CEPI / CITPA
- Heavy duty – CEPE
- Polymeric Coatings on glass – CEPE?
- Coatings on metal foil – FPE / CEPE
- Passivation coatings on metals - APEAL
- SOL-GEL Non-stick coatings – FEC
- Others

Industry response - 5

- Each FCM sector applies the common format for Material Specific Guidelines for Conformity Testing
- Scope
  - Uses
- Definitions
- Material Specific Properties to be considered when testing this class of FCM
  - Brief outline as to why plastic testing guidelines may be inappropriate

Industry response – 6

- Test Procedures
- Evaluation of test results
- Annexes
  - Annex 1: Reasons why plastic guidelines are not suitable for this class of FCM.
  - Annex 2: References
- The amount of detail in each sector's chapters will very considerably, e.g. silicones are relatively specific to bake ware, whereas adhesives cover the majority of adhesives with different issues for different adhesives.

Industry response - 7

- During meetings, different FCM sectors found that other sectors had similar problems with some simulants, times or temperatures.
- There is still debate as to whether to tackle the subject substrate by substrate or material by material e.g. baking paper as paper or as flouro-polymers, silicones etc.
- Some examples of the issues and proposed solutions follow.

Issues with 3% Acetic Acid for Overall Migration

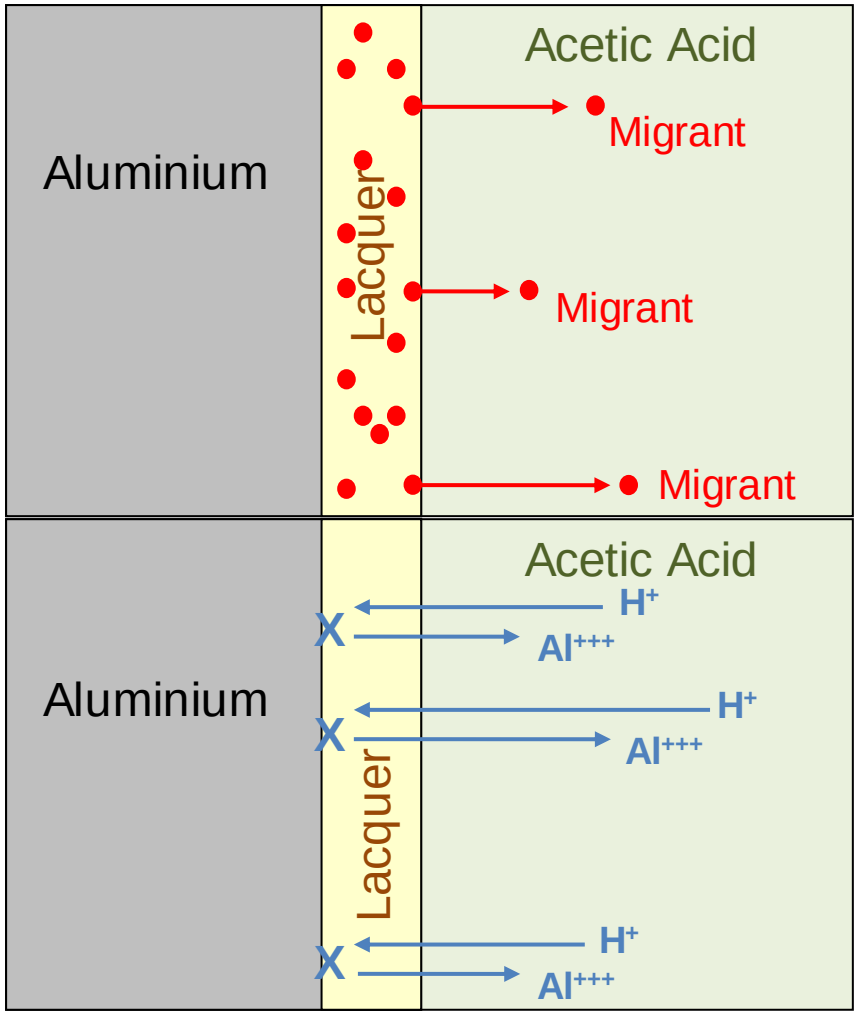
3% Acetic Acid – 1

- A general issue common to many FCM sectors is the use of 3% acetic acid for overall migration (OM).
- OM is NOT a measure of safety, but of inertness.
- Acetic acid corrodes aluminium, either as a coated substrate or foil layer in a multi-layer FCM.

3% Acetic Acid - 2

- Two quite distinct processes are happening during the test
  - True **Migration** – a physical transfer of substances from the **organic** lacquer into the food simulant
  - **Corrosion** – a chemical reaction between acetic acid and the aluminium foil/substrate causing the formation of aluminium acetate and the release of aluminium ions into the food simulant

Schematic Illustration



3% Acetic Acid – 3

- High test results can be obtained, because when the simulant is evaporated to dryness and the residue weighed, the residue is largely aluminium acetate salts
- This increases the weight of the residue, for example:
  - Aluminium molecular weight = 27
  - Aluminium triacetate molecular weight = 204
- Hence up to 87% of the measured result is due to the simulant, not to the aluminium (ions) released.

3% Acetic Acid – 4

- For FPE the overall migration from coated aluminium foil is their biggest issue.
- Coatings for rigid metal packaging also have a major issue with 3% acetic acid.
- The use of citric acid has been proposed, but this has the drawback that in practice it is not volatile enough for a gravimetric determination of overall migration by evaporation.

3% acetic acid – 5

- Proposals being investigated for FPE and coated rigid metal packaging include:
  - 3% acetic acid being used for the extraction, BUT only the chloroform soluble organic material is weighed.
    - Extract in 3% acetic acid, then mix chloroform with the extract, separate the phases and then evaporate the chloroform soluble part to dryness before weighing. Need to confirm that all of the organic material is extracted.
  - For can coatings the use of stainless steel panels and silver foil is being investigated in order to validate chloroform approach.

Issues with Olive Oil and Silicone Elastomers

Silicone Elastomers - 1

- Silicones are exempted from (EU) N° 10/2011 given that elastomers have different physico-chemical properties compared to plastics
- Problems for silicone elastomers arise mainly with compliance testing for baking moulds

Silicone Elastomers - 2

- Olive / vegetable oils contain components penetrating into the silicone elastomer matrix, which results in an overestimation of migration compared to real food. The same applies to its substitutes iso-octane and 95% ethanol.
- Absorbed oil must be removed by soxhlet extraction with a non-polar solvent, which may in turn cause additional extraction of components from the silicone, thereby skewing the results further from reality.

Silicone Elastomers - 3

- A proposed solution is to use Tenax which does not penetrate the silicone elastomer matrix.
- Tenax was an accepted substitute under Directive 97/48/EC but its use as a substitute under 10/2011, lacks clarity.
- Tenax also overestimates migration compared to normal bake ware (e.g. muffins, marble cake) but if the reduction factor of 5 is applied, the results are comparable to standard bake ware.

Silicone Elastomers - 4

- The most important criteria to determine suitability of silicone moulds for food contact is the limit for volatile substances (0.5%) as mandatory according to Recommendation XV of the BfR and the French legislation.
- In appropriately post-cured materials meeting the limit for volatiles <0.5% the majority of migrants consist of cyclic siloxanes with Mw >1000Da which according to present knowledge, are not expected to endanger human health.

Issues with 50% Ethanol

50% Ethanol - 1

- Some polyester based coatings show delamination and swell when tested under severe time/temperature conditions (e.g. 2 h 130 °C) with simulant D1 (50 % ethanol)
- The same polymeric coatings do not show any physical changes when they are in contact with milk products under equivalent sterilisation conditions.
- Overall migration tests and most specific migration tests cannot be carried out in milk products instead of simulants.

50% Ethanol - 2

- Proposal: Compliance of coatings which are not resistant to 50 % ethanol can be demonstrated by

  - decreasing testing time and/or temperature to a level where no physical changes of the coating film occur

  or

  - migration testing with simulant A (10 % ethanol) and D2 (vegetable oil) rather than 50 % ethanol

Relationship between simulant results and those in foodstuffs

Can Coating – example of food – 1

- A coating for metal packaging was needed for a range of foodstuffs with varying oily characteristics, from vegetables in oil to water/oil emulsions.
- Rather than use various simulants, including oil, 95% ethanol was used, as a worst case simulant to cover the range.
- 95% ethanol was an alternative to oil in Directive 97/48/EC, and although not in 10/2011, it is in revised Warenwet which would apply to coatings on rigid metal packaging.
- Customer also requested 95% ethanol.

Can Coating – example of food – 2

- The standard test protocol for 95% ethanol was 4 hrs @ 60º C (for sterilisation at 120 - 130º C), followed by 10 days at 40º C.
- Under the new conditions of 10/2011 and the Warenwet, the storage temperature is now 10 days @ 60º C, if these 'rules' are applied to non-plastics, which is common practice.
- Test results using 95% ethanol gave cause for further investigation.

Can Coating – example of food – 3

- The results of extraction of a monomer into 95% ethanol, 10 days @ 60ºC, were compared to the levels in an aqueous / oily emulsion (D1 simulant in 10/2011) which was in packaging surrounded only by that coating.
- For comparison extraction into acetonitrile and hexane/acetone (1/1) (both 24 hrs @ ambient), gave values between 9 and 16 µg/6 dm^2.

Can Coating – example of food - 4

- The foodstuff was industrially processed and stored for 6 – 12+ months.
- 4 different batches of coating and packaging were used.
- The surface area to volume ratios (S/V) of the packaging was double the EU assumption of 1kg/6 dm^2 - given as corrected concentration using actual S/V ratios.

Can Coating – example of food - 5

- Foodstuff industrially filled and processed < LOD of 70 µg/kg.
- Results of extraction into 95% ethanol.

package sample	storage conditions	conc. (µg/6 dm^2)	corrected conc. µg/kg
1	1 day at 60°C	105	210
	10 days at 60°C	370	740
2	1 day at 60°C	128	256
	10 days at 60°C	386	772
3	1 day at 60°C	133	266
	10 days at 60°C	394	788
4	1 day at 60°C	139	278
	10 days at 60°C	318	636

Can Coating – example of food - 6

- Clearly, the results in 95% ethanol are completely different to that in food.
- Whilst hydrolysis maybe occurring in 95% ethanol, it is not in the presence of the foodstuff.
- Reaching a conclusion about the safety of the coating using the extraction data would give rise to arguably unnecessary concerns about the safety of the coating.

Acknowledgements

- All members of this task force for their inputs.
- In particular, for this presentation, Christa Burger, John Dixon, Beate Ganster, Catherine Simoneau, Richard Whitaker, Ulrich Nehring, Michelle Callow.

THANK YOU FOR YOUR ATTENTION