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Fibre-reinforced plastic composites — Declaration of raw material characteristics — Part 2: Additional requirements for resin, curing systems, additives and modifiers

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Contents

Page

Foreword

This document (prEN xxxx-2:2010) has been prepared by Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by NBN.

This document is a working document.

prEN xxx consists of the following parts, under the general title *Fibre-reinforced plastic composites — Declaration of raw material characteristics*:

— Part 1: *General requirements*

— Part 2: *Additional requirements for resin, curing systems, additives and modifiers (this part)*

— Part 3: *Additional requirements for fibres*

— Part 4: *Additional requirements for fabrics*

— Part 5: *Additional requirements for core* materials

1 Scope

This part of the standard specifies the minimum information to be declared for resins, curing systems, additives and modifiers to be used for the manufacturing of composites products.

These specific declaration requirements are in addition to the general requirements given in part 1 of this standard (i.e. EN xxx-1).

The declaration includes requirements for the certificate of analysis (CoA). The purpose of the CoA is to verify that material properties and quality conforms to the declared values.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN xxxx-1:20xx, Fibre-reinforced plastic composites — *Declaration of raw material characteristics* — Part 1: *General requirements.*

EN 59, *Glass reinforced plastics — Measurement of hardness by means of a Barcol impressor*

EN ISO 62, *Plastics — Determination of water absorption (ISO 62:2008)*

EN ISO 75-2:2004, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics, ebonite and long-fibre-reinforced composites* (ISO 75-2:2004)

EN ISO 178, *Plastics — Determination of flexural properties (ISO 178:2001)*

EN ISO 291, *Plastics — Standard atmospheres for conditioning and testing* (ISO 291:2008)

EN ISO 527 (all parts), *Plastics — Determination of tensile properties*

EN ISO 584, *Plastics — Unsaturated polyester resins — Determination of reactivity at 80 °C (conventional method)* (ISO 584:1982)

EN ISO 1183 (all parts), *Plastics — Methods for determining the density of non-cellular plastics*

EN ISO 2114, *Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value* (ISO 2114:2000)

EN ISO 2555, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method* (ISO 2555:1989)

EN ISO 2592, *Determination of flash and fire points — Cleveland open cup method* (ISO 2592:2000)

EN ISO 2719, *Determination of flash point - Pensky-Martens closed cup method* (ISO 2719:2002)

EN ISO 2811 (all parts), *Paints and varnishes — Determination of density*

EN ISO 2884 (all parts), *Paints and varnishes — Determination of viscosity using rotary viscometers*

EN ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content (ISO 3251:2008)*

EN ISO 3521, *Plastics — Unsaturated polyester and epoxy resins — Determination of overall volume shrinkage (ISO 3521:1997)*

EN ISO 3838:2004, *Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods (ISO 3838:2004)*

EN ISO 4629, *Binders for paints and varnishes - Determination of hydroxyl value - Titrimetric method* (ISO 4629:1996)

EN ISO 4630 (all parts), *Clear liquids — Estimation of colour by the Gardner colour scale*

EN ISO 15512:2009, *Plastics — Determination of water content (ISO 15512:2008.*

ISO 760, *Determination of water — Karl Fischer method (General method)*

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale)*

ISO 3105, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO 14848, *Plastics – Unsaturated-polyester resins — Determination of reactivity at 130 degrees C*

ASTM D93, *Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester*

ASTM D1135, *Test Methods for Chemical Analysis of Blue Pigments*

ASTM D1218, *Standard Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids*

ASTM D2196, *Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer*

ASTM D2583, *Standard Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor*

ASTM D3278, *Standard Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus*

ASTM D4835, *Specification for Propylene Glycol Monomethyl Ether Acetat*e

ASTM D6420, *Test Method for Determination of Gaseous Organic Compounds by Direct Interface Gas Chromatography-Mass Spectrometry*

ASTM E298, *Standard Test Methods for Assay of Organic Peroxides*

ASTM E1473, *Standard Test Methods for Chemical Analysis of Nickel, Cobalt, and High-Temperature Alloys*

ASTM UOP523, *Glycol and Carom Solvent Distribution in Mixtures by GC*

DIN 16945:1976, *Testing of resins, hardeners and accelerators, and catalyzed resins*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
accelerator
substance used in small proportions which accelerate the break down of the initiator to achieve a sufficient rate of generation of free radicals to start and increase the chemical cross-linking reaction rate of an unsaturated resin.

3.2
additive/modifier
substance added to a resin to improve or modify one or more properties (i.e. UV stability, surface tensions, air release etc.).

3.3
Barcol hardness
hardness value obtained by measuring the resistance to penetration of a sharp, spring-loaded steel point with an instrument called the Barcol Impressor. The value can be used as an indicative measure of the degree of cure of a thermosetting resin

3.4
cure
process of chemically converting an unsaturated polymeric composition into a more stable three-dimensional net-work by cross-linking.

3.5
curing system
initiator, promoter, accelerator and inhibitor, all related to the chemical reaction of the resin.

3.6
flash point
the lowest temperature at which a combustible liquid will give off a flammable vapour that will burn momentarily by ignition near the surface of the liquid or within the vessel used.

3.7
geltime
interval of time, in connection with the use of synthetic thermosetting resins, extending from the introduction of an initiator a catalyst into a liquid resin system until the interval of gel formation/solidification.

3.8
inhibitor
substance used in small proportion to suppress a free radical chemical reaction

[EN ISO 472:2001]

3.9
initiator
substance, used in small proportion, that starts a chemical reaction, for example, by providing free radicals

[EN ISO 472:2001]

3.10
monomer
a relatively simple substance, usually containing carbon and of low molecular weight, which can react to form a polymer by combination with itself or with other similar molecules or substances.

NOTE To be distinguished from the term reactive monomer, which within the context of unsaturated polyester/vinyl ester is defined as a substance with unsaturated groups which can undergo cross-linking reactions with the unsaturated polymers through the curing reaction.

3.11
promoter
substance used in small proportion to increase the reaction rate of a free radical chemical process (reactants, plus other additives)

[EN ISO 472:2001]

3.12
resin
<thermosetting materials> the uncured liquid material that is used as matrices to bind together the reinforcement materials in a fibre-reinforced composite product.

3.13
styrene
a colourless liquid produced from the catalytic dehydrogenation of ethylbenzene which is easily polymerized by exposure to light, heat or a peroxide initiator. Is the main reactive monomer used in unsaturated polyester/vinyl ester materials.

3.14
unsaturated polyester
thermosetting resins of polyester molecules, dissolved in a reactive monomer (e.g. styrene) capable of copolymerisation with the polyester molecules. The polyester molecules are condensation products of polyols (e.g. ethylenglycol, dipropylenglycol, glycerol) and saturated/unsaturated difunctional carboxylic acids/anhydrides (e.g. terephathalic acid, phathalic anhydride, fumaric acid, maleic anhydride) and/or cyclic unsaturated hydrocarbons (e.g. DiCycloPentaDiene). At least one of the acids/anhydrids is unsaturated (usually maleic anhydride/fumaric acid is used).

3.15
vinyl ester resins
thermosetting resins of vinyl ester molecules, dissolved in a reactive monomer (e.g. styrene) capable of copolymerisation with the vinyl ester molecules. The vinyl ester is a product of the esterification reaction of any epoxy resin (e.g. Novolac, Bisphenol A epoxy) with an unsaturated monocarboxylic acid (e.g. methacrylic acid).

3.16
viscosity
a measure of the resistance of flow due to internal friction when one layer of fluid is caused to move in relationship to another layer.

NOTE In the context of thermosetting resins for the FRP-industry, viscosity is one of the important parameters that express the ability of a resin to flow in reinforcements and release entrapped air often formed during the application a production process.

3.17
water absorption
amount of water absorbed by a material under specified test conditions.

NOTE The conditions may be immersion in water or exposure to a humid atmosphere, in the latter case the process is also referred to as water vapour absorption.

[EN ISO 472:2001]

4 Content of a declaration

A declaration for the resins, curing systems, additives and modifiers shall consist of information given in part 1 and part 2 (this part) of this standard, i.e. EN xxx-1 and EN xxx-2.

5 Additional declaration requirements

5.1 General

The additional requirements for resins, curing systems, additives and modifiers are given below.

All declaration requirements, i.e. requirements in EN xxx-1 and this part (i.e. EN xxx-2), and application dependant requirements as agreed between manufacturer/supplier and customer, shall be declared by the manufacturer as information to the customer, and the following apply:

- if the property given has reference to a test standard or test method, this test standard or test method shall be used;
- the values given shall be in accordance with the test standard given;
- the tolerances shall be given. If the tolerances are stated in the test standard these apply, if not these shall be specified;
- the clarification of the terms nominal, minimum and maximum test values is given in Annex A;
- if the test environment is not clearly stated in the specific test standard, the standard atmosphere conditioning and testing shall be carried out in accordance with EN ISO 291;
- the manufacturer shall be responsible for the performance and results of all tests required for the declaration;
- the declaration is for the delivered material and not for its constituents.

5.2 Declaration for resin

5.2.1 Declaration for unsaturated polyester and vinyl ester resin

5.2.1.1 Properties of liquid resin as delivered to customer

The additional declaration requirements for liquid resin are listed below. The requirements shall be given in accordance with the test standards stated in Table 1 and Table 2 and including the tolerances.

The following additional declaration requirements a) to f) apply for all liquid resins independent of application:

a) Identification (name/number/code used by the manufacturer for identification purposes);

b) Density [kg/m^3];

c) Viscosity [mPa·s] (cP);

d) Acid number [mgKOH/g];

e) Non-volatile content [wt.%];

NOTE Defines the amount of solid content, from which styrene content in the material can be calculated.

f) Flash point [°C].

The following additional declaration requirements g) to l) apply for liquid resin for specific application:

g) Geltime [min.] [s];

1) Defines the geltime of the resin, at a given temperature and with a given curing system. To be given as arithmetic mean value of ≥ 2 tests within 10 % of mean value with a tolerances.

h) Reactivity [min or s] and [^oC];

i) Maximum recommended laminating thickness [mm];

NOTE Amount and type of initiator shall be given in connection to recommended laminate thickness applied wet in wet. Wet in wet is defined by impregnation of all layers through the thickness prior to curing

j) Colour;

k) Water content [wt.%];

l) Chemical thickening (i.e. valid for SMC resins).

5.2.1.2 Properties of cured non-reinforced resin (clear cast)

Clear cast test sample shall be produced according to Annex B.

The additional declaration requirements for cured non-reinforced resin are listed below. The requirements shall be given in accordance with the test standards stated in Table 3 and including the tolerances.

The following additional declaration requirements a) to n) apply for all cured non-reinforced resins independent on application:

a) Heat distortion temperature (HDT) [°C];

NOTE Defines at which temperature the cured resin deflects a specific amount with a specified load applied.

b) Tensile strength [MPa];

c) Tensile Modulus [MPa] or [GPa];

d) Tensile strain at break [%];

e) Flexural strength [MPa];

f) Flexural modulus [MPa] or [GPa];

g) Barcol hardness;

h) Water absorption [wt.%] or [mg/test specimen];

i) Density [kg/m^3];

j) Defines the density of the cured resin.

k) Volumetric shrinkage [%];

l) Curing system name, number and code (i.e. the exact curing system is to be given);

m) Mixing ratio [wt% and/or vol.%];

n) Post cure schedule – temperature as function of time [ºC and h].

5.2.2 Declaration for epoxy resin

(The additional declaration requirements for epoxy resin are in progress.)

5.3 Declaration for curing systems

5.3.1 Properties of resin initiator

The additional declaration requirements for resin initiators are listed below. The requirements shall be given in accordance with the test standards stated in Table 4 and Table 5 and including the tolerances.

The following additional declaration requirements a) to h) apply for all resin initiator independent on application:

a) Identification (name/number/code used by the manufacturer for identification purposes);

b) Type of initiator;

c) Total active Oxygen content [wt% and /or vol.%];

d) Flash point [°C];

e) Viscosity (nominal) [mPa·s] (cP);

f) Density [kg/m^3];

g) Recommended mixing ratio [wt% and /or vol.%]

h) Defined the recommended mixing ration of the initiator

The following additional declaration requirements i) to m) apply for resin initiator for specific application:

i) Hydrogen peroxide (H_2O_2) content [wt% and /or vol.%];

j) Water content [wt.%];

k) Salt content [ppm];

l) Glycol content [wt.%];

m) Methyl ethyl ketone (MEK) content [wt.%].

5.3.2 Properties of resin accelerator/promoter

The additional declaration requirements for resin accelerators/promoters are listed below. The requirements shall be given in accordance with the test standards stated in Table 6 and including the tolerances.

The following additional declaration requirements a) to i) apply for all resin accelerator/promoter independent on application:

a) Identification (name/number/code used by the manufacturer for identification purposes);

b) Type of accelerator/promoter;

c) Metal content [wt.%] (valid for accelerators only);

d) Active ingredient content [wt.%];

e) Solvent type.

f) Flash point [°C];

g) Viscosity [mPa·s] (cP);

h) Density [kg/m3] - (g/ml);

i) Recommended mixing ratio [wt% and /or vol.%].

5.3.3 Properties of resin inhibitor

The additional declaration requirements for resin inhibitors are listed below. The requirements shall be given in accordance with the test standards stated in Table 7 and including the tolerances.

The following additional requirements a) to f) apply for all resin inhibitor independent on application:

a) Identification (name/number/code used by the manufacturer for identification purposes);

b) Type of inhibitor;

c) Active ingredient content [wt.%];

d) Solvent type;

e) Flash point [°C];

f) Recommended mixing ration [wt.% and/or vol.%].

5.4 Declaration for resin additive/modifier

The additional declaration requirements for resin additives/modifiers are listed below. The requirements shall be given in accordance with the test standards stated in Table 8 and Table 9 and including the tolerances.

The following additional requirements a) to g) apply for all resin additive/modifier independent on application:

a) Identification (name/number/code used by the manufacturer for identification purposes);

b) Type of additive/modifier;

c) Total active ingredient content [wt.%];

d) Flash point [°C];

e) Density [kg/m3];

f) Refractive index;

g) Recommended mixing ratio [wt.% and/ or vol.%].

The following additional declaration requirements h) to p) apply for resin additive/modifier for specific application:

h) Colour;

i) Acid Value;

j) Amine value;

k) OH-number;

l) Non-volatile content [wt.%];

m) Solvent type;

n) Water content [wt.%];

o) Viscosity [mPa·s];

p) Particle size/Particle size distribution [µm] (applicable for particulate fillers only).

6 Content of Certificate of Analysis (CoA)

6.1 General

Certificate of analysis (CoA) shall be delivered on request. The certificate shall verify that the delivered batch is within the agreed number of declared values. The relevant test methods used to obtain batch data shall be the same as used for the declaration.

Each sheet of the CoA shall be clearly marked with identification and batch number.

6.2 CoA for resins

6.2.1 CoA for unsaturated polyester and vinyl ester resins

6.2.1.1 CoA for liquid resin

The certificate of analysis for liquid resin, with reference to relevant items given in 5.2.1.1, is given in Table 1 for the application independent liquid resin properties and in Table 2 for the liquid resin properties for specific application.

Table 1 — CoA properties for liquid resin independent of application

Ref. no 5.2.1.1	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
b)	Density			[kg/m^3] [g/cm^3]	EN ISO 2811	O
c)	Viscosity, high Shear Viscosity, low Shear			[mPa·s] (cP)	EN ISO 2884 ASTM D2196	●
d)	Acid Number			[Mg KOH/g]	EN ISO 2114	O
e)	Non Volatile content			[%]	EN ISO 3251	●
f)	Flash point			ºC	ASTM D3278 EN ISO 2592	O

[a] ● - Compulsory
O – Optional

Table 2 — CoA properties for liquid resin for specific applications

Ref. no 5.2.1.1	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
g)	Gel time			[min. or sec.]	None	O
h)	Reactivity Ambient Temp. Reactivity Elevated Temp. SMC			[min./sec and ºC]	DIN 16945:1976, 6.2.2.2 EN ISO 584 ISO 14848	O
j)	Colour				ISO 2211 EN ISO 4630	O
k)	Water content			[wt.%]	ISO 2211 EN ISO 4630	O

[a] ● - Compulsory
O – Optional

6.2.1.2 CoA for Cured non-reinforced properties (clear cast)

The certificate of analysis for non-reinforced resin (clear cast), with reference to relevant items given in 5.2.1.2, is given in Table 3 for the application independent non-reinforced resin (clear cast) properties.

Table 3 — CoA properties for application independent cured non-reinforced resin (clear cast)

Ref. no 5.2.1.2	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
a)	Heat Distortion Temp.			[ºC]	ISO 75-2 Method A	O
b)	Tensile strength			[MPa]	ISO 527	O
c)	Tensile modulus			[MPa] [GPa]	ISO 527	O
d)	Elongation			[%]	ISO 527	O
e)	Flexural strength			[MPa]	ISO 178 Method A	O
f)	Flexural modulus			[MPa] [GPa]	ISO 178 Method A	O
g)	Barcol Hardness			Units	EN 59	O

(To be continued)

Table 3 — CoA properties for application independent cured non-reinforced resin (clear cast) (continued)

Ref. no 5.2.1.2	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
h)	Water Absorption			Wt.% or mg/test specimen	EN ISO 62	O
i)	Density			[kg/m^3]	ISO 1183	O
j)	Volumetric shrinkage			[%]	EN ISO 3521	O
k)	Curing system name, number and code					O
l)	Mixing ratio			[wt% and/or vol.%]		O
m)	Post cure schedule			[ºC and h]		O

[a] ● - Compulsory
O - Optional

6.2.2 CoA for epoxy resins

The certificate of analysis for epoxy resin is in progress.

6.3 CoA for curing systems

6.3.1 CoA for resin initiator

The certificate of analysis for resin initiator, with reference to relevant items given in 5.3.1, is given in Table 4 for the application independent resin initiator properties and in Table 5 for the application dependent resin initiator properties.

Table 4 — CoA properties for application independent resin initiator

Ref. no 5.3.1	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
c)	Total Active Oxygen content			[wt%] [vol.%]	ASTM E298	●
d)	Flash point			[°C]	ASTM D3278	●
e)	Viscosity			[mPa·s] (cP)	ISO 3105	O
f)	Density			[kg/m^3] [g/cm^3]	EN ISO 3838	O

[a] ● - Compulsory
O - Optional

Table 5 — CoA properties for a resin initiator for specific applications

Ref. no 5.3.1	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
h)	Hydrogen peroxide (H_2O_2) content			[wt%] [vol.%]	Modified ASTM E298	O
i)	Water content			[wt.%]	EN ISO 15512 by Karl Fisher titration	O
j)	Salt content			[ppm]	ASTM D1135 (conductivity method)	O
k)	Glycol content			[wt.%]	ASTM D4835 or ASTM UOP523	O
l)	Methyl ethyl ketone (MEK) content			[wt.%]	ASTM D6420	O

[a] ● - Compulsory
O - Optional

6.3.2 CoA for resin accelerator/promoter

The certificate of analysis for resin accelerator/promoter, with reference to relevant items given in 5.3.2, is given in Table 6 for the application independent resin accelerator/promoter properties.

Table 6 — CoA properties for application independent resin accelerator/promoter

Ref. no 5.3.2	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
c)	For Accelerators: Metal Content	Cobolt Other		[wt.%]	ASTM E1473	●
d)	For Promoters: Active ingredient content/Aniline content			[wt.%]	ASTM E298	●
f)	Flashpoint			[°C]	ASTM D3278	O
g)	Viscosity			[mPa·s] (cP)	ISO 3105,	O
h)	Density			[kg/m^3] [g/cm^3]	EN ISO 3838,	O

[a] ● - Compulsory
O - Optional

6.3.3 CoA for resin inhibitor

The certificate of analysis for resin inhibitor, with reference to relevant items given in 5.3.3, is given in Table 7 for the application independent resin inhibitor properties.

Table 7 — CoA properties for application independent resin inhibitor

Ref. no 5.3.3	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
c)	Active ingredient content			[wt.%]		●
e)	Flashpoint			[°C]	ASTM D3278	O

[a] ● - Compulsory
O - Optional

6.4 CoA for resin additive/modifier

The certificate of analysis for resin additive/modifier, with reference to relevant items given in 5.4, is given in Table 8 for the application independent resin additive properties and in Table 9 for the application dependent resin additive properties.

Table 8 — CoA properties for application independent resin additive/modifier

Ref. no 5.4	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
c)	Total active ingredient content			[wt.%]		●
d)	Flash point			[°C]	EN ISO 2719	O
e)	Density			[kg/m^3] [g/cm^3]	EN ISO 2811	O
f)	Refracting index				ASTM D1218	O

[a] ● - Compulsory
O - Optional

Table 9 — CoA properties for resin additive/modifier for specific application

Ref. no 5.4	Property	Declared value with tolerances (% or range)	CoA Test result	Unit	Test method	CoA Content [a]
h)	Colour				EN ISO 4630	O
i)	Acid value				EN ISO 2114	O
j)	Amine value				DIN 16945	O
k)	OH-number				EN ISO 4629	O
l)	Non-Volatile content			[wt.%]	EN ISO 3251	O
n)	Water content			[wt.%]	EN ISO 15512 or Modified Karl Fisher titration/ ISO 760	O
o)	Viscosity			[mPa·s] (cP)	EN ISO 2555	O
p)	Mean particle size / Particle size distribution			[µm]		O

[a] ● - Compulsory
O - Optional

Annex A
(informative)

Clarification of nominal, maximum and minimum test values

A.1 General

The requested properties as given in this part of the standard EN xxx (i.e. EN xxxx-y), are given as nominal values with upper and lower limits, maximum values and/or minimum values. The purpose of this annex is to clarify the nomenclature and the associated values.

A.2 Clarification of nominal value

Nominal value means that the properties of the material will have a mean value equal to the declared value. The mean value is based on all measured values of all batches of the manufacturer. The material delivered to the customer is normally only a fraction of the totally produced material. This may result in that the actual value on the property of the delivered material has a mean value lower or higher than the declared value. However, the mean value of the property of the delivered material shall not be outside the declared tolerance limits in order to be delivered according to the declared properties. These tolerances include inaccuracy in the measurement itself.

A.3 Clarification of minimum and maximum value

Minimum value and maximum value means the mean value minus (-) and plus (+) two standard deviations, respectively.

The minimum and maximum value is based on all measured values of all batches of the manufacturer. The delivered materials have a 97,6 % probability of being within the minimum and maximum values, thus having 2,4 % probability of being outside the minimum and maximum value. This implies that part of or all of the delivered material may belong to the fraction which is outside the minimum and maximum values. In order to avoid this, an agreement between the manufacturer and customer stating that no material shall have properties above or below the declared minimum and maximum values, i.e guaranteed minimum or maximum values.

A.4 Guaranteed minimum and maximum value

Guaranteed minimum and maximum value mean that no delivered material has properties below or above these values. When destructive testing is needed the mean value of the tested material (within the same batch) shall be within the declared minimum and maximum value for that particular test if guaranteed values have been agreed. In case a guaranteed value is not agreed, a sample test may have any value and still be within the specified range.

A.5 Extent of testing

The extent of testing involved for a CoA has to be agreed between the supplier and the customer. For a resin material it is normal to obtain the properties from the batch. For core and fibre material, several of the tests are destructive tests and have to be performed on undelivered material.

For core plates, destructive testing for mechanical properties can be performed in the lower range of density, while non destructive tests, (dimensions and density) may be performed on every plate or a selection.

The extent of testing of fibre material is similar as for core material, where typical values are obtained from one or more bobbins or an agreed selection.

Annex B
(informative)

Non-reinforced/clear cast mouldings of unsaturated polyesters and vinyl ester

B.1 Principle

Non-reinforced/clear cast mouldings of unsaturated polyesters and vinyl esters are made to test mechanical and physical properties.

Air voids, curing stresses and other possible defects in the moulding will influence the final results negatively and give results which are not correct. Moulding thixotropic resins and/or Low Styrene Emission (LSE) resins as delivered may also cause reduced moulding quality, as the thixotropic agent and paraffin wax can cause “micro-air bubbles” inclusions which could further influence the tested properties. Therefore it is always recommended to use only non-thixotropic and non-waxed resins for moulding to test mechanical properties.

The method described is to ensure that a non-reinforced moulding of unsaturated polyester resin and/or vinyl ester resin is produced with as few defaults as possible. The method also contains a guidance for specimen selection for reporting average values.

B.2 Apparatus

The apparatus used for non-reinforced/clear cast:

- two clean glass plates, 450 mm x 300 mm (see A.3.2);
- flexible rubber gaskets, E-profile 5 mm thickness x 10 mm width (see A.3.2);
- aluminium spacer sheets with thickness of 4 mm.

B.3 Procedure

B.3.1 Curing system

Select a cobalt-MEKP (methyl ethyl ketone peroxide) system inhibited with 4-tert-butylcathechol (TBC) to a geltime of 30 minutes to 50 minutes at 23ºC. Avoid using curing systems that can give curing stresses, for example too reactive curing systems.

After having carefully stirred in separately the accelerator and the peroxide, let the mix deairate/degas for approximately 20 minutes.

B.3.2 Mould preparation filling

Thoroughly clean two glass plates with acetone and soap water and clean thoroughly with clean water prior to waxing.

Prepare the glass plates (450 mm x 300 mm) with an appropriate release wax giving good release properties (for example Mirror Glaze 88).

Let the waxed glass plates “dry/cure” minimum 2 hours after polishing.

Prepare a gasket, normally a rubber gasket E-profile with thickness 5 mm and width 10 mm. Place the prepared gasket around the edges of one of the prepared glass plates, and put the other glass plate on top.

Insert a 2 mm x 2 mm or a 1 mm x 4 mm aluminium spacer sheet to ensure even and correct thickness.

Tighten the glass mould with an appropriate clamping system to a mould opening of 4 mm ± 0,1 mm.

The prepared mould is placed in a certain angle (i.e. for example 60º to 80º).

Before the resin is moulded, it might be necessary to dilute with styrene. Generally approximately 200 mPas at 23ºC for orthophtalic resins and 300 mPas at 23ºC for isophtalic and vinyl ester resins are used.

Before filling the mould, the resin shall be filtered, preferably through a 100 microns filter.

The resin is gently poured into mould, so that no air bubbles are created .

B.3.3 Curing

After reaching the exothermic peak the clamps are slightly loosened.

The resin is cured 18 hours to 24 hours at room temperature (23ºC) in the mould.

B.3.4 Demould

The clear casting is placed horizontally between two aluminium plates or glass plates, protecting the casting on both sides with Mylar film. For flexible resin mouldings a soft paper tissue can be used instead of the Mylar to reduce possible surface defects. The top plate shall act as a weight.

B.3.5 Post curing

Place the demoulded non-reinforced/clear cast in a heating oven. It is important that the oven is “cold” when starting the post curing process. The cool down period must be slow and the moulding shall not be removed from the oven at temperatures above 40°C.

If no other post curing cycle is specified, 24 hours at 60ºC plus 3 hours at 10ºC above the anticipated heat deflection temperature (HDT) of the resin is used.

If the highest post curing temperature exceeds 90ºC - 100ºC, one shall post cure step-wise to avoid stresses in the casting. Use intervals of 25ºC -30ºC. See example bellow:

EXAMPLE

- 90ºC in 1/2 hour;
- increase to 120ºC in 1/2 hour;
- increase to 150ºC in 3 hours;
- cooling.

B.4 Test specimens

The following is prepared to give guidance to selection of specimens to be used for testing/reporting.

When the moulding is demoulded and post cured as described in A.3.4 and A.3.5 above, the test specimens are cut according to the test standards to be used.

The cutting edges of the specimens shall be polished.

A visual judgement of the test specimens shall be carried out to exclude those who contain air-entrapments or voids from the casting and those who have been damaged or have edge failures from the cutting or machining of the specimens.

Test specimens that differ considerably from the rest of the specimens concerning width and thickness are excluded.

After testing the specimens the individual results shall be evaluated. The test specimens that are giving large deviations compared to the others shall be examined. If these test specimens contain air voids or other visual defects, they shall be excluded and shall not be used in the final calculation/report. If other individual specimens show large deviation in results they shall be evaluated if they shall be part of the final calculation/report.

B.5 Quality assurance and control

Typically 6, 9 or 12 tensile test specimens and 6 or 9 flexural specimens are made.

If the number of specimens that are excluded according to the above procedure are larger than 1/3, i.e more than 3 specimens of a total selection of 9 specimens, a new non-reinforced/clear cast moulding shall be made.

The absolute minimum of repeats to be used for calculation/reporting is 5 specimens.

Any deviation from the above description/method shall be documented and compared for equal results.

Bibliography

[1] EN ISO 472:2001, Plastics — Vocabulary (ISO 472:1999)