



EUROPEAN ADHESIVE ENGINEER

MODULE 8.2

RAW MATERIALS CONTROL



8.2 Raw Materials Control

Objectives:

- ✓ Know and control aspects regarding raw materials, such as:
 - Supplier certification
 - Manufacturing system qualification
 - Incoming specifications
 - Testing

- ✓ Guarantee the correct and necessary storage of material

Raw Materials Importance [1]

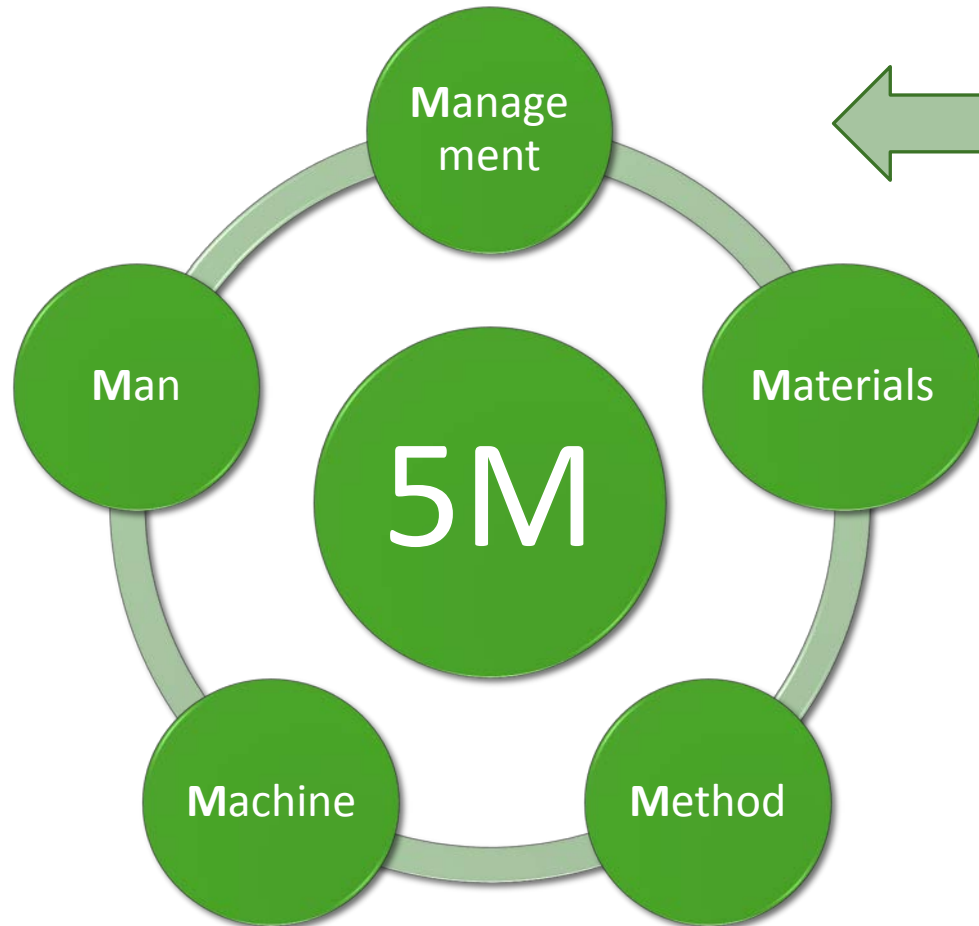


It is very important

- Selection
- Storage
- Handling
- Testing

Raw
Materials

Quality Control of Adhesives [1]



The most important element to control the quality of adhesives!

Defined as the actual conducts to carry out the whole cycle of:

- ✓ "Plan"
- ✓ "Do"
- ✓ "Check"
- ✓ "Action"

PDCA

Supplier Certification ^{[2],[3]}

- The supplier certification acts as a part of a larger strategy of the supplier quality management, within a quality management system.

- This component of a total quality management system assures that a supplier's product is:
 - Produced
 - Packaged
 - Shippedunder a controlled process that results in consistent conformance to the requirements

- ➔ It allows the organization to manage its relationship with its suppliers, working as a single entity.

Supplier Certification ^[3]

- The supplier certification specifies minimum requirements expected from the supplier to be eligible for working
 - It is important for all suppliers of any materials in the bonding process:
 - Adherents
 - Consumables
 - Adhesive
 - Fixed equipment

- This process aims at a **continuous improvement**.

Supplier Certification ^[3]

The Supplier Certification is important because:

□ It allows:

- ✓ the process of identifying suppliers which can meet the requirements of the company
- ✓ to gather a fair estimate of the potential of the suppliers, thus enabling judicious and safe decisions in terms of investing
- ✓ avoidance of some processes such as inspection.
- ✓ the development of supplier capabilities, leading to improvements in efficiency.

□ It regulates and builds consistent methods for managing the suppliers

Supplier Certification ^[3]

The Supplier Certification is important because:

- ❑ It establishes more open and stronger relationships

- ❑ It facilitates enhanced communication and better sharing of information

- ❑ It helps:
 - ✓ the organization serve its customers better
 - ✓ achieve corporate improvement objectives through collaboration

Manufacturing ^[4]

- Maintaining good control over the manufacturing process will ↑ the probability of achieving a defect-free joint → this is particularly important in the case of adhesive strength for which no satisfactory NDT method currently exists.
- As adhesive manufacturers design and provide adhesives which meet the demand of the market, raw material makers should provide appropriate products to adhesive makers, understanding and considering their needs.
- There is widespread irrefutable evidence that improved quality during manufacture greatly ↓ the cost of making and inspecting the parts ⇒ If there are no defects to be found, there are no costs incurred by fixing them.

Quality-control in Adhesive Bonding [5]

Incoming Specifications: necessary part of a quality control program



statement of the requirements that the adhesive, sealant, or process must meet in order to be accepted for use



A product specification is an agreement between supplier ↔ user.



BUT compliance with a specification doesn't mean that the adhesive or sealant will perform perfectly in service!

- It only means that the product complies with the specification.
- Bonding or sealing specifications should not only account for the adhesive or sealant, but they should also define the adherends and the ancillary processes for preparing the adherends and the joint assembly.

Quality-control in Adhesive Bonding [5]

- The **product specification writer** must try to put into the specification the **requirements** that, if met, will provide the **greatest likelihood of success**.



should be standard tests that are agreed upon by both the supplier and the user

- The tests:
 - should be indicative of how the adhesive is used in production and how the finished joint is to be used in service
 - are directly related to the specific application
 - should not be used simply because they are standard test methods or have been used in the past
- Specifications from other sources (e.g., ASTM, military, etc.) may be used if they are applicable.

Quality-control in Adhesive Bonding [5]

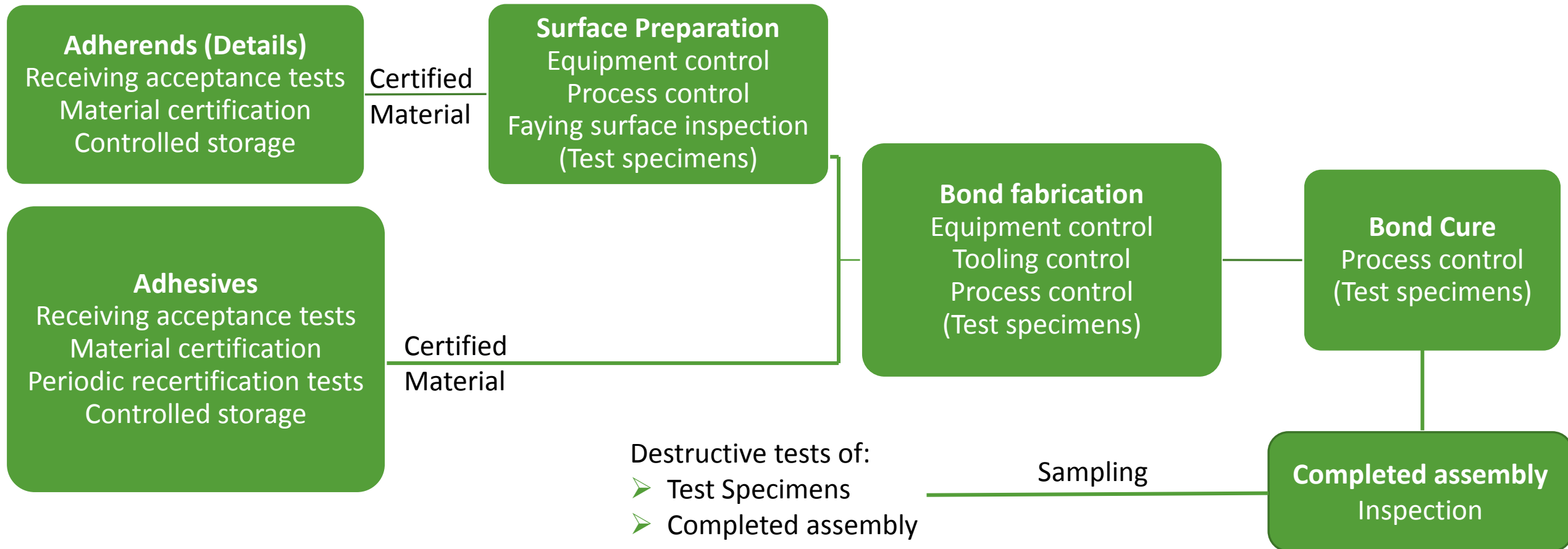
- Specifications may require ≠ categories of testing
 - For example, there may be extensive series of tests that are required for initial verification or qualification of the adhesive and supplier.
- These tests would be used to approve a certain product at the onset. Other receiving tests may be used to verify the consistency of the product from lot to lot.

Format of a typical
specification



- Title
- Scope
- General requirements
- Performance requirements
- Test methods
- Controls
- Reference documents
- Approved source list

Quality-control in Adhesive Bonding [6]



Incoming Material Control [6]

- Quality control begins on the reception of **raw materials** such as adhesives and catalysts.
- The purchase order ordinarily defines the required quality properties of this material.
- This is accomplished by an actual statement of requirements or by what is called out in the material specifications.
- The inspection requirements are normally specified in the material specifications as **Quality Acceptance Tests** or as **Receiving Inspection Requirements**.

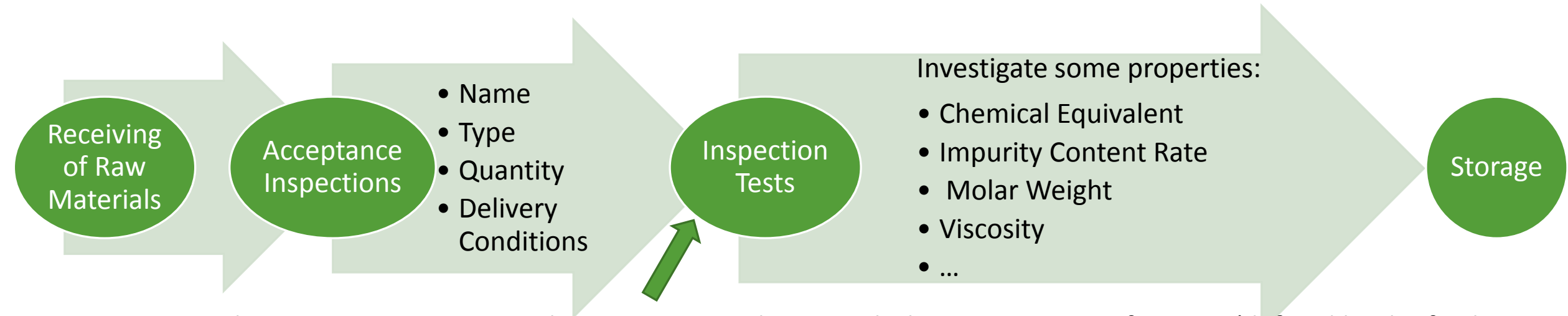
Incoming Material Control [6]

Containers

The 1st inspection requirement is normally the condition of the container. The following items should be checked when inspecting the container:

- ❑ **Damage** : Physical damage to a container of film adhesive can rupture its sealed wrapper, allowing moisture, dirt, etc., to reach and contaminate the adhesive. Damage can render a pail of liquid measure unusable in automatic measuring equipment.
- ❑ **Leakage** : Leakage of liquid adhesive components can change the ratio of the catalyst to the base resin if premeasured kits are involved. It can also result in the receipt of less material than the purchaser needs and is paying for.
- ❑ **Identification** : Identification of a container should include:
 - ✓ product number
 - ✓ manufacturer 's instructions for use
 - ✓ date of manufacture
 - ✓ batch or lot nr
 - ✓ shelf life
 - ✓ recommended storage conditions
 - ✓ manufacturer's name
 - ✓ safety precautions

Quality Control Procedures for Raw Materials [7]



Aim: Required on any incoming materials, to prove compliance with the incoming specifications (defined by the final customer or manufacturer).

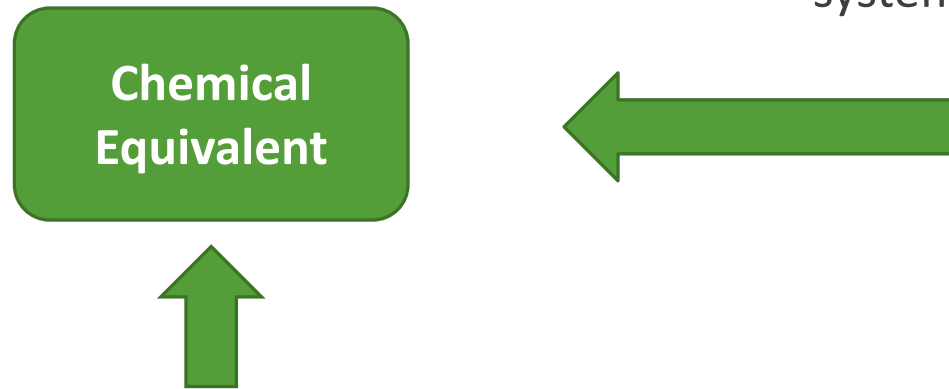
If these tests are carried out by **suppliers**: users should check the specification documents indicating the test results BUT tests by users are still important to make sure the quality of their products!

⇒ Capabilities of users to conduct tests are indispensable to decide the types and sequences of tests, which should be discussed and agreed with the suppliers testing the materials.

Quality Control Procedures for Raw Materials

Inspection Tests [7]

This tests must be adequate to the process or according to a specification and supplier's quality system



For raw materials of **reactive adhesives**, the chemical equivalent values are very important to specify the reactivity, and often tested.

Epoxy equivalents for epoxy resins, and functional group equivalents for hardeners are experimentally determined.

Methods to measure the epoxy equivalents are described by industrial standards:

- ✓ International organization for standardization (ISO) 3001
- ✓ Japan industrial standards (JIS) K 7236
- ✓ American society for testing and materials (ASTM) D 1652.

Quality Control Procedures for Raw Materials

Inspection Tests [7]



Adhesives for **electronic applications** are sensitive to impurities.

For instance, inclusion of conductive particles often induces problems of insulation breakdown.

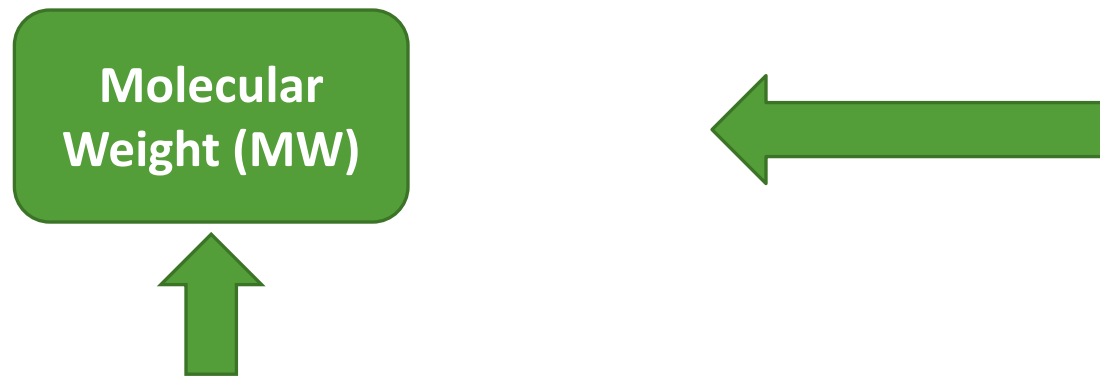
Chlorine inclusion can cause corrosion of metal parts such as electrodes made of oxygen-free copper foils on flexible circuit boards that can react with chlorine in adhesives for bonding the foils and the boards, yield dendrites, and lead to insulation breakdown.

Since the phenomena occur at very low density of chlorine, the content rates should be checked by standards such as:

- ✓ ISO 21627
- ✓ JIS K 7243.

Quality Control Procedures for Raw Materials

Inspection Tests [7]



Since main components of adhesives are usually monomers of ↓ MW:
⇒ The performance of the adhesives isn't influenced by the variation of the MW
⇒ The measure of the MW of the components isn't important.

BUT, for **PSAs**, raw materials such as rubbers and tackifiers have relatively higher MW than those of adhesives

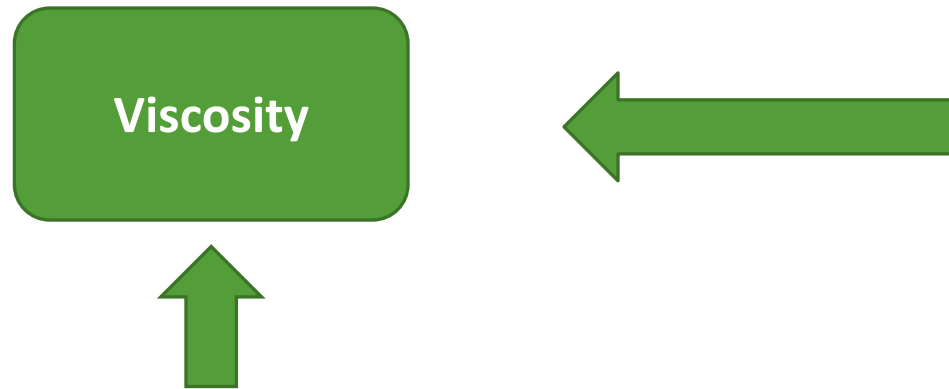
⇒ The variations lead to deviations of the final performances!

The MW of the raw materials for PSAs are often measured by experimental methods such as :

- ✓ End Group Determination
- ✓ Colligative Property Measurements
- ✓ Light Scattering
- ✓ Ultracentrifugation
- ✓ Viscosity Measurements
- ✓ Gel Permeation Chromatography (GPC)

Quality Control Procedures for Raw Materials

Inspection Tests [7]



Viscosities of raw materials are very important because they influence the:

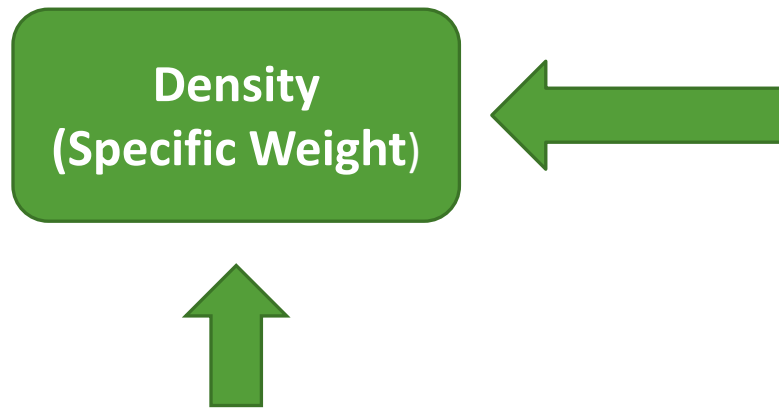
- Properties of Final Adhesive Products
- Production Process

Methods for measuring viscosities of liquid raw materials are included in the following standards:

- ✓ ISO 2555
- ✓ ISO 3219
- ✓ JIS K 7117
- ✓ ASTM D1084

Quality Control Procedures for Raw Materials

Inspection Tests [7]



Densities and specific weights are basic material properties and should be measured to check errors of adhesive production

The measurement methods of specific weights are defined by standards:

- ✓ ISO1183
- ✓ JIS K7112
- ✓ ASTM D792
- ✓ ISO 3675
- ✓ JIS-K2249

Densities are basically equivalent to specific weights, and the selection depends on the nature of the material:

- Density (liquid mass/volume) → Liquids
- Specific Weights → Solids

The density of a liquid can be measured with:

- Pycnometers
- Specific gravity cups, whose inner volumes are known

Quality Control Procedures for Raw Materials

Storage [7]

❑ Circumstance controls are very important for raw materials in storage

❑ The most influential condition on material degradation is **T**



- Air conditioners for warehouses
- Refrigerators
- Freezers

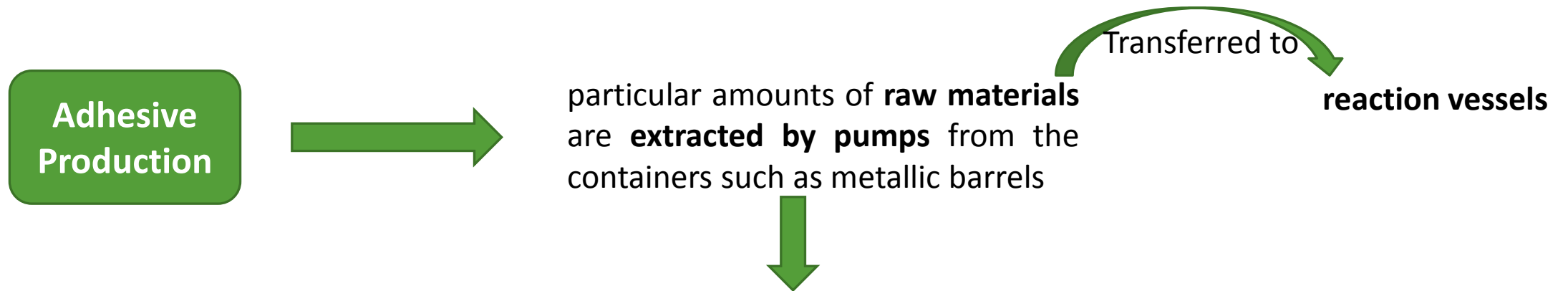
should be appropriately selected for the required T conditions of raw materials

❑ Although **humidity** and **UV irradiation** are lesser influential than T, **protection methods** have to be taken:

- In storage
- In processing of adhesive products

Quality Control Procedures for Raw Materials

Storage [7]



- Some materials such as **isocyanate resins** are very **sensitive to humidity**, and solidify when the containers are kept open

⇒ The **time limits of opening containers** should be specified and kept by workers

- **Consuming up materials soon after the containers are opened** is a good idea ✓

⇒ Proper plans for the production of adhesives can contribute to the quality control of raw materials

Quality Control Procedures for Raw Materials

Storage [7]



If **containers** for raw materials are **dirty and contaminated**
⇒ The cleanliness of the clean rooms may decrease!

⇒ The **containers should be stored in clean conditions** in order to prevent them from contaminations

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

- When adhesives are shipped after the processing, users of raw materials become supplier of products
- **Inspection** should be done to the **adhesive** products to prove the performances.
- **Specification sheets** should be issued for users if required.
- Sampling tests of industrial adhesives are rare unlike household adhesives. Tests of each production lot are usual.

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Inspection tests before shipments of adhesive products:

- Adhesion Strength
- Tackiness – very important for PSAs
- Hardness
- Viscosity
- Density
- Volume Shrinkage
- Thermal Expansion
- Shelf Life
- Pot Life
- Gelation Time
- Water Resistance
- Chemical Resistance
- Weather Resistance
- Flame Retardance
- Electric Conductivity
- Colour
- Optical Properties
- Amount of VOC Emergence

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Shelf Life of an adhesive

- Term during which adhesive can be stored without undesirable physical and chemical changes
 - Depends on the storing condition of the adhesive
- Should be indicated with the storage condition such as:
 - T
 - Humidity
 - Light.
- Some adhesives are sensitive to oxygen or humidity ⇒ The shelf lives of the adhesives should be stored with unopened container during a term, then the **viscosities or joint strengths are experimentally measured.**
- The procedure is denoted in test standards such as **ASTM D 1337**

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Pot Life (Working Life) of an adhesive

- Time length in which an adhesive can be applied and used to join after the adhesive comes to an applicable condition.
 - For instance, 2-part epoxy adhesives gradually cure and harden after the mixing of the parts, and become too stiff to be applied after their pot lives, decreasing the bond strength.
 - For moisture curable adhesives, the pot lives start when the adhesives are extracted from the containers.
- Pot lives are determined measuring the **viscosities or joint strengths** of adhesives **with respect to time**.
- The procedures are denoted in some standards:
 - **ISO10364**
 - **JIS K 6870**
 - **ASTM D 1338**

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Curing Shrinkage

➤ **Reactive adhesives** such as epoxy and acrylic resins **may induce shrinkage stresses** that **reduce** often the **bonding strengths**.

⇒ The volume shrinkage should be measured by standards such as **JIS K6911**, in which adhesives are cured in a mold and the dimension of the adhesive is measured to calculate the shrinkage ratio.

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Curing Rate

- Curing rate is important information for users of adhesives.

- However, ordinary adhesives cure gradually and sometimes reach to the final strength within a few days.
 - ⇒ The time to the final strength is less important than the time when de-molding or handling of the joints can be possible, because the total time of bonding processes highly depends on the de-molding or handling time.

 - In contrast, a too short curing time (\approx short pot life) spoils the workability.

- ⇒ Curing rates should be measured using many specimens such as **lap shear joints with respect to time.**

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Strength and Hardness

- The **strength** of the adhesive in bulk is not so important to be often measured BUT sometimes it is necessary to **verify the specification**.
- For the strength test of adhesives in bulk, dog bone specimens for tension or rectangular specimens for bending are used.
- In contrast, **hardness** of adhesives in bulk can be used as an **index of curing condition**, and is measured in terms of **quality control**.

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Strength and Hardness

- Hardness measurements include scratch tests and indentation tests, and the latter, such as the Shore test, is often utilized for adhesives in bulk.
 - A Shore hardness tester has a small diamond half sphere equipped at the tip of a hammer, and the hammer hits the surface of a specimen.
 - Hardness is calculated from the height of the hammer rebounding after the collision.
- **A too soft adhesive ⇒ under-cure or mixture of air bubbles**
- **A too hard adhesive ⇒ over curing**

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Glass Transition Temperature (T_g)

- Important parameter for the quality control of adhesive formulation.
- Related to the **maximum usable T 's of adhesives.**
- Adhesives are softened and become viscoelastic around the T_g .

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Glass Transition Temperature (T_g)

- The T_g of an adhesive can be measured with:
 - **DSC** (Differential Scanning Calorimetry): calculated from the absorbed or emitted **energy** measured
 - **TMA** (Thermo Mechanical Analyzer): calculated from the **deformation**
 - **DMA** (Dynamic Mechanical Analyzer): calculated from the **fluctuation of viscoelastic parameters**
 - ❖ BUT the values from the methods are slightly different.

⇒ The definition of T_g is quite ambiguous.

- There is another similar T called **softening point** measured by penetration or bending tests using **TMA**. This is very **close to T_g** in value

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Other Properties

- Nowadays, additional functions become important and the performance should be tested.
- For instance, since the use of Pb solder is nowadays prohibited by the restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS), conductive adhesives are used instead.
- For the adhesives, **electric conductivity and shear resistance** are vital, and their tests, besides other conventional tests, should be conducted
 - ⇒ Some adhesives developed in the future may have novel functions that conventional adhesives don't have, and new test methods and standards should be required to check the functions.

Quality Control Procedures for Raw Materials

Storage: Inspection for Shipment [7]

Other Properties

- From the **environmental point of view**, measurements of **VOC** emergence and **dismantlability** of adhesives will become + important in the future

- Since qualities of raw materials influence considerably the performances of adhesive products, **the materials should be tested** appropriately by **test standards** depending on the types of materials.

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