

The production of rigid polyurethane foam

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1. Introduction

Rigid polyurethane foam is currently one of the best thermal insulating materials available. As a result, thermal insulation is a key feature of almost all its applications. The possibility of combining rigid polyurethane foam with different facing materials to produce composites also gives it an important role as a construction material. The principal areas of use for rigid polyurethane foam are:

Domestic appliances

- Thermal insulation for domestic and commercial refrigerators and freezers, hot water tanks

The building industry

- Sandwich panels with rigid facings as wall and roofing panels
- Insulating boards with flexible facings for roofs, walls, ceilings and floors
- Insulating and construction material as cut-to-size pieces from slabstock
- Spray-in-place foam for insulation and sealing

Industrial thermal insulation

- Insulation of tanks and containers, pipelines, district heating pipes and cold stores

The automotive industry

- Thermal insulation of refrigerated vehicles for road and rail including containers

The production of rigid polyurethane foam for applications in the building industry will be described in the following chapters.

Rigid polyurethane foam has a number of particular advantages:

- it can be produced in a wide range of densities.
- it adheres to various facings without the use of adhesives.
- it can also be produced in complex cavities.

The production of rigid polyurethane foam requires two main liquid components - a polyol and a polyisocyanate - and a blowing agent. The blowing agent is usually added to the polyol together with further auxiliary components such as activators (reaction accelerators), foam stabilizers and flame retardants. The polyaddition reaction that takes place when the polyol and polyisocyanate are mixed together results in macromolecules with urethane structures (**polyurethanes**).

During the reaction a considerable amount of heat is released which is used partly to evaporate readily volatile liquids (blowing agents). As a result, the reaction mix is expanded to form a foam.

Various quantities of water are normally added to the polyol. The water reacts with the polyisocyanate to form **polyurea** and **carbon dioxide**, which serves as a co-blowing agent but can also be the sole blowing agent.

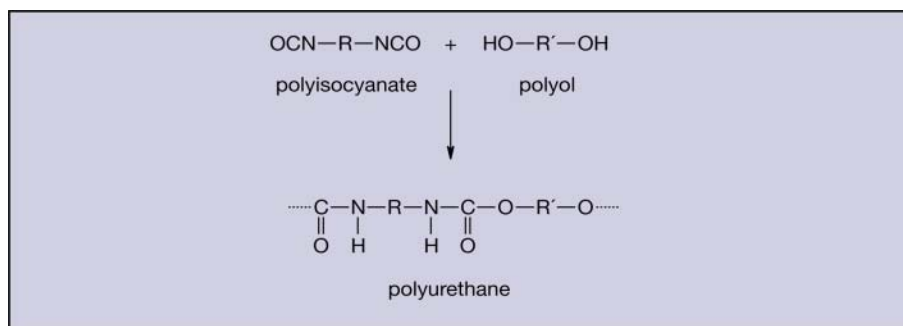


Fig. 1: Polyurethane reaction formula

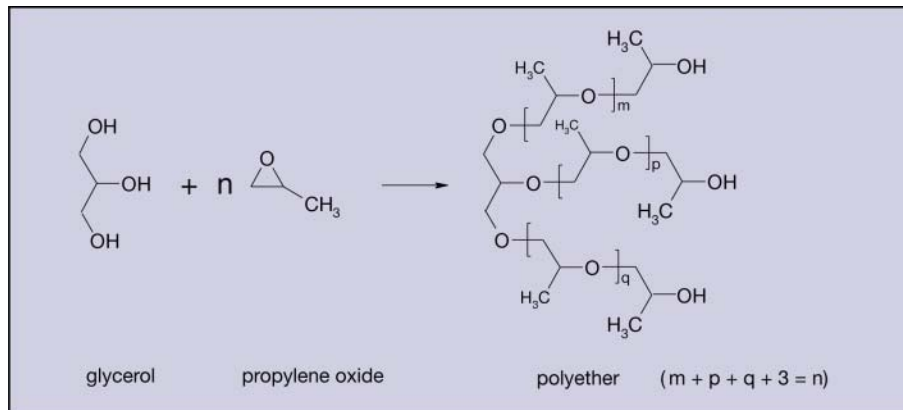


Fig. 4: Polyether production using a trihydric alcohol as an example

Polyester polyols are obtained by reacting polybasic carboxylic acids (for example phthalic, terephthalic or adipic acids) with glycols, glycerol and similar

polyalcohols. Polyols with ester structures are formed with water being eliminated.

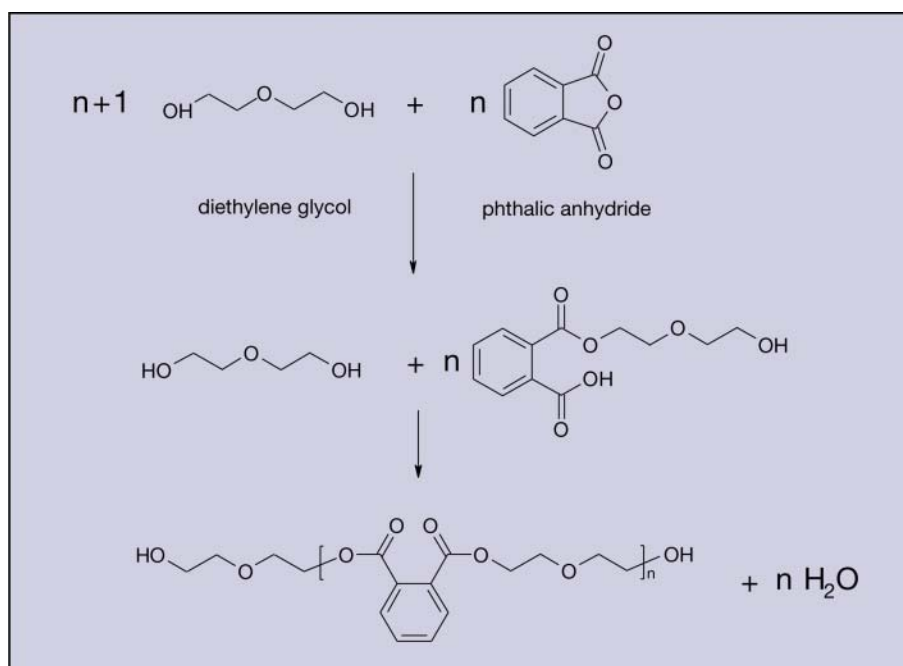


Fig. 5: Principle of polyester production

There are no general rules governing the use of polyether polyols and polyester polyols. The two types are often used in combination, with the final selection being determined by individual requirements. In order to achieve the flame retardant effect required for the foam, it is often advisable to use them in conjunction with aromatic polyester polyols, for example based on phthalic acid. High viscosity

and low functionality limit the use of ester polyols in many cases.

The **functionality** of a polyol indicates the number of hydroxyl groups per molecule. If the polyol is a mixture of components with different functionalities, the **average functionality** is given. Parts of the molecule which can undergo reactions, such as, for

example, the hydroxyl groups, are called **functional groups**. A measure of the hydroxyl group content is the **hydroxyl** or **OH-value**.

To select a polyol, information on the hydroxyl (OH)-value, viscosity and water content is necessary in addition to the chemical nature. The acid value may also be important with polyester polyols.

2.2 Polyisocyanates

Chemical compounds with **isocyanate groups** (NCO-) as functional groups are known as isocyanates.

Isocyanate groups are based on nitrogen, carbon and oxygen. Isocyanates based on **MDI** (= **methylene diphenylene diisocyanate** known as diphenylmethane diisocyanate or diisocyanato-diphenylmethane) are used almost exclusively for producing polyurethane foam. These are mixtures of MDI (mainly **4,4'-diisocyanato-diphenylmethane** with an isomeric 2,4'-diisocyanato-diphenylmethane content) and higher molecular components. As a molecular unit is repeated in the structure of these higher molecular components, the isocyanate mix is also called **polymeric MDI (PMDI)** or **MDI polymer**.

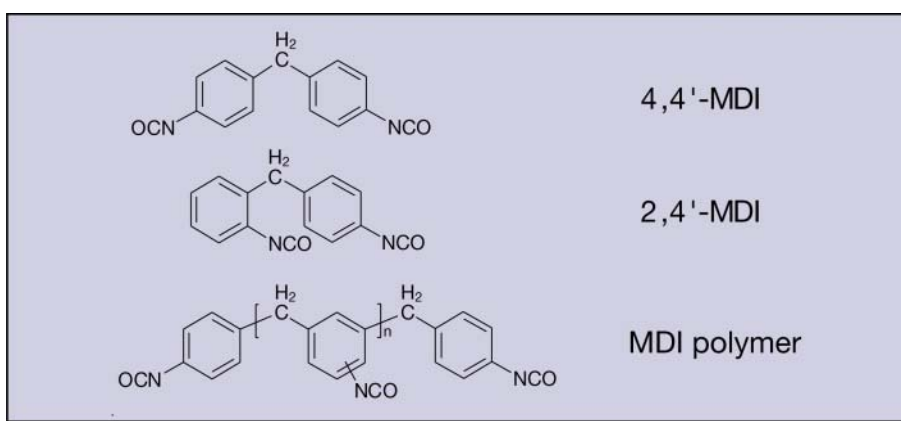


Fig. 6: Diphenylmethane diisocyanate, MDI polymer

While MDI is strictly difunctional, i.e. has two NCO groups, the higher molecular PMDI-components contain three and more NCO groups. PMDI is therefore known as a polyisocyanate. The average functionalities of the conventional PMDI types are about 2,5 to 3,2.

PMDI prepolymers should be mentioned at this point although they only play a small part in the production of rigid polyurethane foam. The PMDI involved is one in which some of the NCO groups have been made to react by the addition of polyol. Compared to the starting PMDI, the NCO content is therefore lower and the viscosity significantly higher. With the aid of prepolymers, problems can be averted and certain effects achieved: the quantity of heat released during production of the foam is reduced and the compatibility of polyisocyanate with polyol and the structure of the macromolecule are influenced.

Polyisocyanates based on MDI for the production of rigid polyurethane foam are viscous liquids which

are brownish to dark brown in color. They are characterized by **NCO content**, **viscosity** and **acidity**.

Acidity is a measure for the production-dependent acid content which can influence reactivity.

2.3 Blowing agents

As already mentioned, the reaction of isocyanate and water yields carbon dioxide which is used as a blowing agent. This is referred to as a **chemical blowing process**. This process has some disadvantages which is why it only has a subsidiary role in the production of rigid polyurethane foam. The actual blowing process is the **physical blowing process** in which a low-boiling liquid incorporated in the reaction mix is vaporized due to the heat of the reaction.

The blowing agents should dissolve well in the reaction components and the reaction mix, should remain in the closed cells of the foam as a gas and

possess low heat conductivity, be almost insoluble in polyurethane and not plasticize it. They should also be toxicologically and ecologically safe.

The chlorofluorocarbon (CFC) 11 (monofluorotrichloromethane) which was used for a long time met most of these criteria excellently but gave reason for concern from an ecological viewpoint. The **global warming potential (GWP)** of CFC 11 and its potential to damage the ozone layer of the stratosphere (**ODP = ozone depletion potential**) led to the decision to discontinue use (Montreal Protocol of 1987).

Carbon dioxide is soluble in polyurethane and therefore escapes from the foam cells by diffusion if the foam is not made diffusion-tight. As a result of the falling gas pressure in the cell the foam can shrink. The polyurea, which is formed simultaneously with the carbon dioxide, can also cause the foam to become brittle, impairing adhesion to the facings (for example sheet metal).

Nowadays, **hydrocarbons** (pentanes), **hydrogen chlorofluorocarbons (H-CFCs)** and **hydrogen-fluorocarbons (H-FCs)** are used instead of CFC 11. None of these blowing agents are direct replacement products for CFC 11. When using these blowing agents, the polyols and additives have to be adapted to obtain the required foam properties.

Pentanes (n-, cyclo- and iso-pentane) are highly flammable liquids, the vapors of which form explosive mixtures with air. When they are used as blowing agents, certain safety precautions have to be taken with corresponding expenditure (see Technical Information No. 1/1998 "Pentane: An option for the production of CFC- and H-FC-free sandwich

panels" [2]), and the necessary capital investment has to be made. The great advantage of pentanes is their low price. N-pentane is used predominantly for the production of rigid polyurethane foam for the building industry as it is particularly economical and, in contrast to cyclo-pentane, no plasticizer effect is observed. However, n-pentane has only low solubility in the reaction components and its vapor has higher thermal conductivity.

A limited time period was established by the countries signing the Montreal Protocol for the use of H-CFCs, as a reduced, but nevertheless considerable ODP or GWP is ascribed to them. Of the H-CFCs 22, 142b and 141b which can be industrially produced, only H-CFC 141b is still being used. Its advantage is the low thermal conductivity of the gas but its disadvantage is the plasticizer effect.

H-FC 134a, the only product in this category available at the moment apart from 152a, has only limited use. It is incorporated under pressure in liquid form and dissolves only with difficulty in the reaction components. As it evaporates spontaneously when the pressure is released, the reaction mix foams immediately and is therefore difficult to distribute.

H-FC 245fa and 365mfc, which will be available in the foreseeable future, are possibilities for future blowing agents. Both products are liquid at normal pressure and dissolve adequately in the reaction mix.

The most important properties of the above-mentioned blowing agents are shown in the following table.

Blowing agent	Formula	Mol weight	Boiling point °C	Flammability	ODP	GWP	Thermal conductivity (gas) mH/m · K
CFC 11	CCl ₃ F	137.4	23.7	–	1	1	8.5 (25°C)
H-CFC 22	CHClF ₂	86.5	– 40.8	–	0.055	0.36	10.8 (25°C)
H-CFC 141b	CH ₃ –CCl ₂ F	117.0	32.0	flammable	0.11	0.12	10.1 (25°C)
H-CFC 142b	CH ₃ –CClF ₂	100.5	– 9.2	flammable	0.065	0.42	12.9 (25°C)
H-FC 134a	CH ₂ F–CF ₃	102.0	– 26.3	–	0	0.25	13.7 (25°C)
H-FC 152a	CH ₃ –CHF ₂	66.0	– 24.7	flammable	0	0.03	14.3 (25°C)
H-FC 245fa	CF ₃ –CH ₂ –CHF ₂	134.0	15.3	–	0	0.24	13.0 (25°C)
H-FC 365mfc	CF ₃ –CH ₂ –CF ₂ –CH ₃	148.0	40.2	flammable	0	0.21	10.6 (25°C)
n-Pentane	C ₅ H ₁₂	72.0	36.0	flammable	0	0.00044	14.0 (25°C)
i-Pentane	C ₅ H ₁₂	72.0	28.0	flammable	0	0.00037	13.6 (25°C)
c-Pentane	C ₅ H ₁₀	70.0	49.0	flammable	0	0.0004	12.4 (25°C)
CO ₂	CO ₂	44.0	– 78.5 sublimated	–	0	0.00015	16.0 (25°C)

Table 1: Properties of various blowing agents for polyurethane foams

2.4 Activators

Most polyols and polyisocyanates only react with one another at a moderate rate at room temperature. The same is true for the reaction of polyisocyanate with water, so accelerators (activators) are added to the reaction mix. These are usually tertiary amines, organo-tin compounds or alkali salts of aliphatic carboxylic acids which particularly promote isocyanurate formation. The best known products are triethylamine, dimethylcyclohexylamine, dibutyltin dilaurate and potassium acetate. Some of the individual compounds from the large number of activators have very different effects on the reactions described. This can be used to control the progress of the reaction and foaming according to requirements.

2.5 Foam stabilizers

The foam which forms as a result of the developing or evaporating blowing agent is usually unstable and would collapse if the reaction continued without the addition of foam stabilizers. Organo-silicon compounds (polyether polysiloxanes) which have a surface-active effect are used almost exclusively as foam stabilizers, but also function as emulsifiers. Foam stabilizers regulate the foam structure, the open- and closed-cell character and the cell size and therefore have a substantial influence on the foam properties.

2.6 Flame retardants

Polyurethanes are organic compounds and, as such, are flammable. In order to delay their ignition and reduce the spread of the flames, an appropriate chemical structure and the addition of flame retarding components are required. As already mentioned, aromatic polyester polyols and polyisocyanurate (PIR) structures, for example, contribute to fire safety. The use of halogen-containing polyols is also usual. Non-reactive additives are trialkyl, tris-halogen alkyl and triaryl phosphates. Triethyl phosphate, tris-chlorisopropyl phosphate and diphenyl-cresyl phosphate are typical examples. Solid, insoluble flame retardants are scarcely used as they are problematical from the processing point of view. However, they could play a more important role in the future.

The existing flammability tests make different demands on the fire performance of the foam (see also Technical Information No. 10/1999, "Current state of European standards on polyurethane rigid foams in the construction industry" [3]). In most cases the measures on offer have to be combined to pass the respective flammability test.

2.7 Bayer AG raw materials

Bayer AG offers raw materials or **raw material systems** for producing rigid polyurethane foams for a wide range of applications. Raw material systems are products with a chemical structure tailor-made for the requirements of the particular application. They can be two-component or multi-component products.

Although a system of this type consists simply of a polyol or a polyol mix with additives and a polyisocyanate, the term "system" is often applied to the polyol, because the application orientation is usually via the polyol and the polyisocyanate remains unchanged. It is also usual for a mixture of polyols and additives to be known as **polyol formulation**. A mixing plant for polyols is called a formulating plant. In practice, all the forms between base polyol and complete polyol formulation are on offer. The most frequent form of a **partial formulation** is a mixture of two and more polyols which are mixed with activators, stabilizers and flame retardants before processing.

The components necessary for producing rigid polyurethane foam are supplied by Bayer AG under the following trade names:

- **Desmodur**[®] polyisocyanate
- **Desmophen**[®] base polyols or base polyol mixtures
- **Baytherm**[®] polyol systems/formulations for non-flame retardant rigid polyurethane foams
- **Baymer**[®] polyol systems/formulations for flame retardant rigid polyurethane foams
- **Desmorapid**[®] activator
- **Disflamoll**[®] flame retardant
- **Levagard**[®] flame retardant

Foam stabilizers can be obtained, for example, from GE Bayer Silicones, Th. Goldschmidt AG, Air Products GmbH, Nitroil Europe Handels GmbH and Witco Corp.

Blowing agents are sold, for example, by Erdölchemie GmbH, Exxon Chemical GmbH and Haltermann (alkanes), Solvay Fluor und Derivate GmbH and DuPont (H-CFCs and H-FCs).

2.8 Storage of raw materials

All raw materials for producing rigid polyurethane foams should be protected from moisture during storage. Polyols are generally hygroscopic and absorb water from the air, so the water content can increase by a few percent in a short period of time and can lead to shifts in characteristic values (see Section 3.1), increased CO₂-generation and therefore lower foam density and reduced foam strength.

Protection against moisture is particularly important in the storage of polyisocyanates. The polyurea which forms during reaction with atmospheric moisture is separated off as a solid so encrustations occur in the storage container and particles are deposited which can block the lines and nozzles. For this reason, air entering the storage container when raw materials are removed should be dry.

Polyurethane raw materials can only be stored for a limited time. While most of the basic components can be stored unmixed in sealed containers for months and years, mixtures (formulations) are likely to undergo a chemical change in a matter of weeks. This is shown by discoloration, increased viscosity, and a reduction in reactivity and foam stability.

High temperatures can sharply reduce the storage stability of the raw materials. Care must be taken in this respect particularly during long periods of transportation and heating caused by solar radiation. As a rule, the raw materials available on the market are adequately stable over fairly short periods of time (days to weeks) at temperatures up to 50 °C. This has to be the case as the temperature of the raw material can often be 50 °C, for example with tanker deliveries. A processing temperature of 20 to 25 °C is recommended in most cases, which means that appropriate cooling, for example by heat exchangers, should be arranged on receipt of the raw materials. Temperature control of the raw materials during prolonged storage is also appropriate. High temperatures can impair the quality,

and low temperatures can impair the processability by making the viscosity too high.

For completeness it should be noted that raw materials containing blowing agents

(H-FC, H-CFC) should generally be stored at temperatures below 30 °C.

As a general rule, the statutory regulations on storing chemical raw materials should always be observed.

2.9 Safety precautions when handling polyurethane raw materials

When handling polyurethane raw materials, the safety precautions described in the product information sheets and product safety data sheets should be observed for safety reasons.

Polymeric MDI (PMDI) is classified as a dangerous substance and requires a hazard warning label. It must be handled with care. An occupational exposure limit has been set which defines the maximum permissible workplace concentration, in the form of gas, vapor or airborne particulates, of a specific chemical or chemicals contained in PMDI. The degree of risk depends mainly on the quantities of isocyanate vapors and aerosols released when processing PMDI. Unless PMDI is to be sprayed, no problems arise when processing at 20 to 23 °C, provided this is done in a well ventilated area. It is however essential to provide adequate exhaust ventilation at each workplace, with the air being drawn away from the personnel handling the product.

Vapors and aerosols of PMDI cause irritation to the eyes and the mucous membranes of the nose, throat and lungs, and may lead to hypersensitivity reactions. Inhalation should therefore be avoided.

Safety goggles, impermeable protective gloves and overalls fastened at neck and wrist should always be worn when handling the product. Contaminated clothing should be removed immediately to prevent further skin contact. A good barrier cream should be rubbed into the hands before starting work. PMDI should be kept away from food, drink and tobacco. Further technical information relating to safety can be found in the Safety Data Sheet.

The precautions and rules described above should always be observed during the production of rigid polyurethane foam.

3. Principles of rigid polyurethane foam production

3.1 Calculating the formulation

For a raw material system according to section 2.7, a processing formulation is usually given which prescribes the proportions in which polyol and polyisocyanate are to be mixed, for example:

100 parts by weight polyol
 x parts by weight activator
 y parts by weight blowing agent
 140 parts by weight polyisocyanate

However, if the aim is to formulate a raw material system from basic components, the proportions have to be calculated. To do this, the hydroxyl number (OH value), water content and isocyanate content (NCO) have to be ascertained. The calculation formulae are derived as follows:

Index

The calculated formulation must ensure that an NCO group of the polyisocyanate is available for each OH group of the polyol. The index shows whether this condition is met. The index is defined as the product of the number of NCO mols per OH mol multiplied by 100.

$$KZ = \frac{\alpha_{\text{NCO}}}{\alpha_{\text{OH}}} \times 100 \quad \text{Equitation 1}$$

When the index is 100, the above condition is met. An index > 100 means an excess of NCO. Formulations for producing rigid polyurethane foam normally have an index between 105 and 125, and those for rigid polyisocyanurate-polyurethane foams an index between 180 and 350. With polyurethane formulations, an index > 100 ensures the complete reaction of the OH group. Excess NCO groups react, for example, while forming allophanate structures.

As α_{NCO} and α_{OH} cannot be directly determined, they are calculated with the aid of the NCO content and the OH value.

Calculation of the OH value and of α_{OH} :

To determine the hydroxyl value (OH value), a gram of the polyol is reacted (esterified) with the anhydride of a dicarboxylic acid and the released hydroxyl-equivalent acid is titrated with potassium hydroxide (KOH).

The hydroxyl value is defined as the necessary quantity of KOH in mg per g of polyol. If the quantity M_{polyol} is used instead of 1 g of polyol, the definition of the OH value is as follows:

$$\text{OHZ} = \frac{M_{\text{KOH}}}{M_{\text{polyol}}}$$

If this equation is not related to mg of KOH but to mMol of KOH, both sides of the equation must be divided by 56.1 (molecular weight of KOH):

$$\frac{\text{OHZ}}{56.1} = \frac{M_{\text{KOH}}}{M_{\text{polyol}} \times 56.1} = \frac{\alpha_{\text{KOH}}}{M_{\text{polyol}}}$$

$$\alpha_{\text{KOH}} = \text{Mole KOH}$$

Since it follows from determining the hydroxyl value that one mol of OH corresponds to each mol of KOH, α_{KOH} and α_{OH} can be equated:

$$\frac{\text{OHZ}}{56.1} = \frac{\alpha_{\text{OH}}}{M_{\text{polyol}}}$$

Hence transformation produces:

$$\alpha_{\text{OH}} = \frac{\text{OHZ} \times M_{\text{polyol}}}{56.1} \quad \text{Equitation 2}$$

Calculation of α_{NCO} :

The quantity of NCO in the polyisocyanate is normally given in % by weight of NCO, being:

$$\% \text{ NCO} = \frac{M_{\text{NCO}}}{M_{\text{polyisocyanate}}} \times 100$$

If the connection between quantities and molarity is taken into consideration in accordance with

$$\alpha_{\text{NCO}} = \frac{M_{\text{NCO}}}{42}$$

42 = molecular weight of NCO

and mol is converted simultaneously to mMol, then one obtains

$$\% \text{ NCO} = \frac{\alpha_{\text{NCO}} \times 42}{1000 \times M_{\text{polyisocyanate}}}$$

Hence, α_{NCO} can be calculated from the transformation:

$$\alpha_{\text{NCO}} = \frac{\% \text{ NCO} \times M_{\text{polyisocyanate}}}{4.2} \quad \text{Equitation 3}$$

Calculation of the polyisocyanate content of a formulation:

If equations 2 and 3 are inserted into equation 1, the amount of polyisocyanate necessary for a specific quantity of polyol at a predetermined characteristic value can be calculated:

$$\begin{aligned} \text{KZ} &= \frac{\% \text{ NCO} \times M_{\text{polyisocyanate}} \times 56.1}{4.2 \times \text{OHZ} \times M_{\text{polyol}}} \\ &= \frac{\% \text{ NCO} \times M_{\text{polyisocyanate}} \times 1336}{\text{OHZ} \times M_{\text{polyol}}} \end{aligned}$$

Transformation gives

$$M_{\text{Polyisocyanat}} = \frac{\text{OHZ} \times M_{\text{polyol}}}{1336 \times \% \text{ NCO}} \times \text{KZ} \quad \text{Equitation 4}$$

This formula makes it possible to calculate the quantity of polyisocyanate necessary at a specific index for a given quantity of polyol given the OH value and the NCO content of the polyisocyanate.

3.2 Preparation of the reaction components

If the polyol component already contains the additives required for foaming, all that is necessary before processing, as with the polyisocyanate, is to bring the temperature to 20 °C, for example. In

most cases the polyol component is supplied without an activator or blowing agent. Both additives then have to be added after temperature control. Care should be taken here to ensure that the blowing agent has also been brought to the correct temperature in advance.

A certain constant temperature is required to ensure a constant reaction and foaming process (see Section 3.3). If flame retardants, water, stabilizers etc. are added to the polyol, time should be allowed for the dissolving process which starts during mixing. This means that once mixing has been completed, the polyol component may possibly not have reached its final quality. Particular attention should be paid to this aspect when mixing in water. A fresh polyol mix can behave quite differently during foaming to a stored mix. This possible behavior of the polyol mix should be determined. To avoid problems, it is advisable to heat the polyol mix briefly to 50 °C without blowing agent or to leave it to stand for 1 to 2 hours.

Similar effects can also occur when the blowing agent is mixed in, but its effect is usually insignificant.

3.3 Reaction mix and foaming

For foam production, the prepared raw materials (i.e. the polyol component and the polyisocyanate component) which have been temperature controlled and provided with additives, are thoroughly mixed together.

The reaction starts after a short period of time and progresses with heat development. The reaction mix is continually expanded by the blowing gases released, until the reaction product reaches the solid state as a result of progressive crosslinkage, the foam structure being retained.

The following stages are characteristic of the reaction and foaming process:

- The **mixing time** or **stirring time** indicates the time needed for mixing the reactants.
- The **cream** and **foaming time** is the time which elapses from the start of mixing of the reactants to the visible start of foaming of the mix. In many cases this can be seen clearly by a color change. With slow reacting mixes this requires practiced observation.

- The **string or fiber time** is the transition of the reaction mix from the liquid to the solid state. It roughly corresponds to the gel point. When this point in time is reached the reaction is about 50 % complete. The fiber time is measured by, for example, a

wooden rod being repeatedly immersed in and removed from the already well expanding reaction mix, and it is determined when the rod draws fibers. Time measurement begins with mixing.

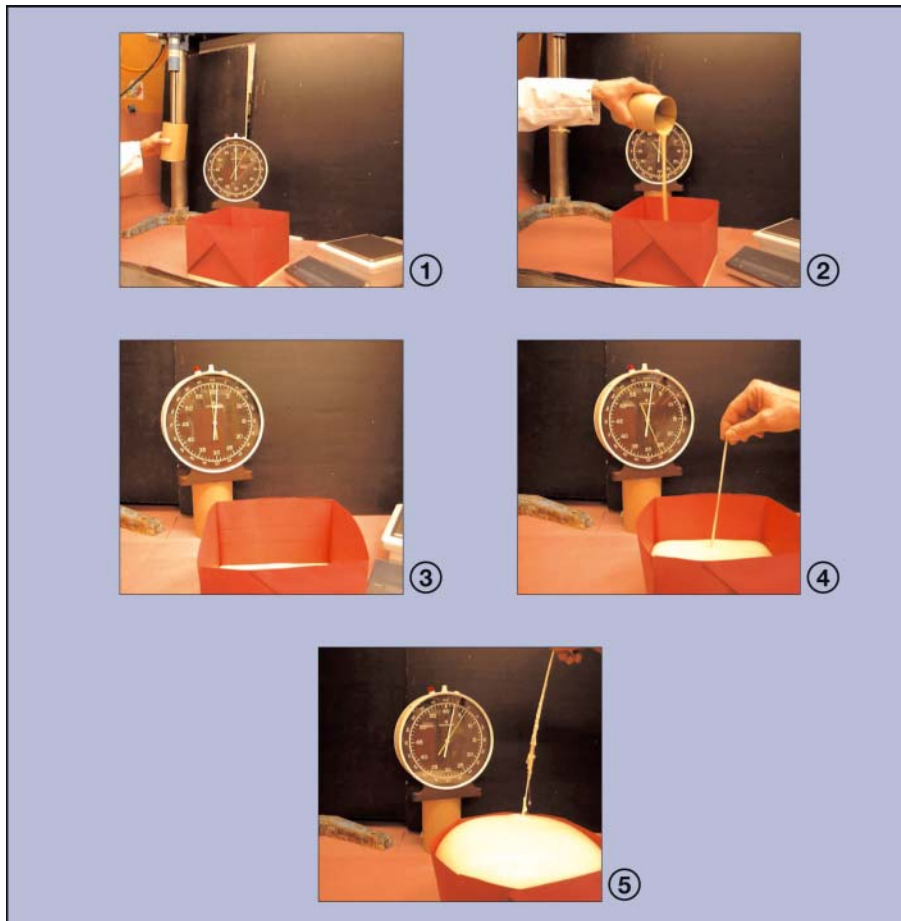


Fig. 7: Laboratory test: Measuring the fiber time

- **Full rise time and tack-free time:**
After fiber time, the speed at which the foam rises slows down. The time from the start of mixing till the end of the optically perceptible rise is called the rise time. The surface of the foam is still tacky when the rise process is complete. By repeatedly testing the foam surface with a wooden rod, the moment of freedom from tack is determined. The time elapsing from the start of mixing to the moment when the surface is no longer tacky is called **tack-free time**.

- **Gas release time:**
Open-cell foams can open their surface after a certain time and release excess blowing gas. The time elapsing from the start of mixing to the start of gas release is called the **gas release time**.

Reaction times are dependent on the temperature of the reaction components, i.e. the reaction mix. The reaction times decrease as the temperature rises. If the fiber time is 100 s at a starting temperature of 20 °C, it reduces to 60 s, for example, at 30 °C. The fiber time gives the surest and most accurate information on reactivity, which is why it is used almost exclusively for establishing the reaction

rate. Comparative data on reactivity constantly have to refer to identical temperatures.

There are various theories about the **development of the foam**. Most are based on **nucleation** (bubble formation) in the development phase. Whether all the cells present in the finished foam are already present in the early development phase is unclear. It can be assumed that supersaturation of the liquid reaction mix with the blowing gas and cavity formation (cavitation) when the raw materials are mixed is responsible for nucleation. The formation of a fine gas dispersion (air, already formed blowing gas) during mixing could also be significant.

In this early foam development phase, the presence of surface-active compounds such as foam stabilizers is decisive.

The dispersed spherical gas bubbles initially grow due to diffusion of the blowing gas. This process lasts until a certain volume is achieved in which the spherical cells are most densely packed in the liquid matrix. If this volume is exceeded, the spherical cells convert to polyhedral cells, mainly pentagonal dodecahedrons. The majority of the polymer liquid is located here in the struts and walls, while thin membranes separate the individual cells from one another. This cell growth is shown in the following diagram.

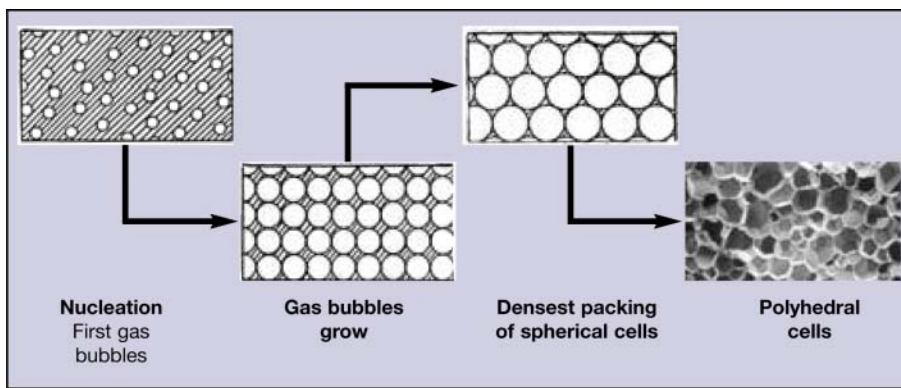


Fig. 8: Cell growth during development of rigid polyurethane foam

The foam reaches its final structure and mass distribution at the end of fiber time. The diagram showing spherical cells and corresponding polyhedral structures is not quite accurate in practice. As the foam has a flowing movement in the expansion phase and the cells hinder each other, they take on a stretched form, i.e. the diameter in the flow direction is greater than in the vertical direction perpen-

dicular to it. Reference is made to **cell orientation**. This cell stretching - which remains after curing if the foam is not prevented from extending - is reflected in the properties of the foam. Thus the compressive strength in the foaming direction can be up to three times that in the direction perpendicular to it.

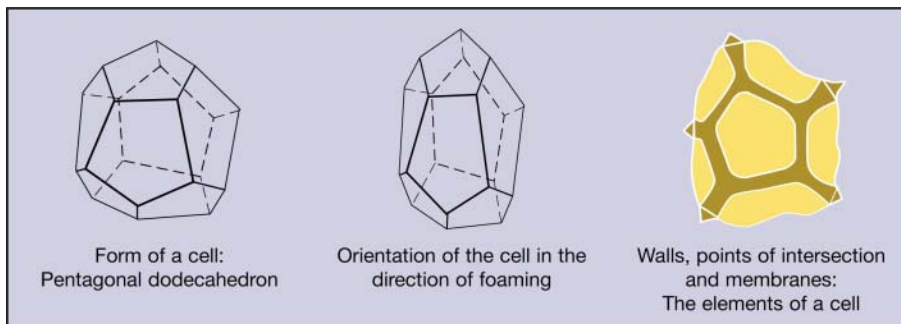


Fig. 9: Structure of a foam cell

The following illustration shows the flow of the reaction mix in the expansion phase and illustrates the effects. Concentric chambers were arranged at the bottom of a beaker and filled to the same level with

variously dyed reaction mixes. To show individual time phases, a row of beakers was charged with increasing quantities of reaction mix.

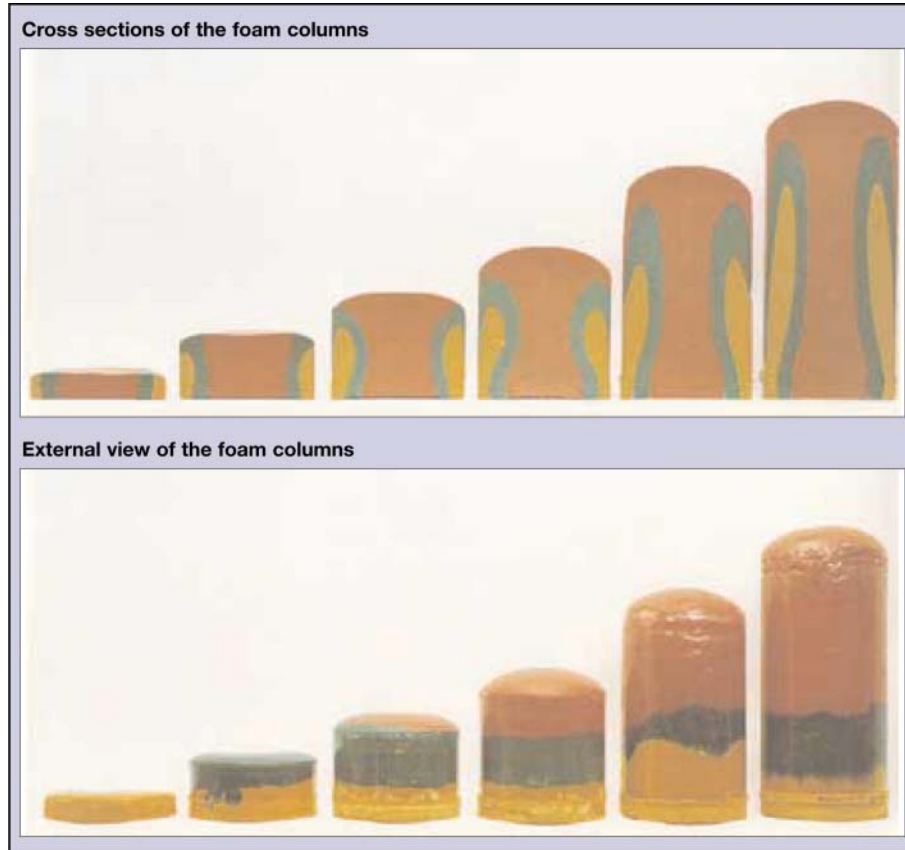


Fig. 10: Cross sections and external views of the foam columns

Temperatures between 120 and 180 °C occur in the foam core depending on the raw material system, and the reactivity, volume and density of the foam. The gas pressure in the cells increases as a result of heating and has to be absorbed by the cell walls and membranes. The time-scale of the expansion in

volume (rise of the foam), the pressure and the temperature during the foaming and curing process is shown in the following graph. A vertically arranged tube was filled with foam from the bottom and the data were measured (see page 30):

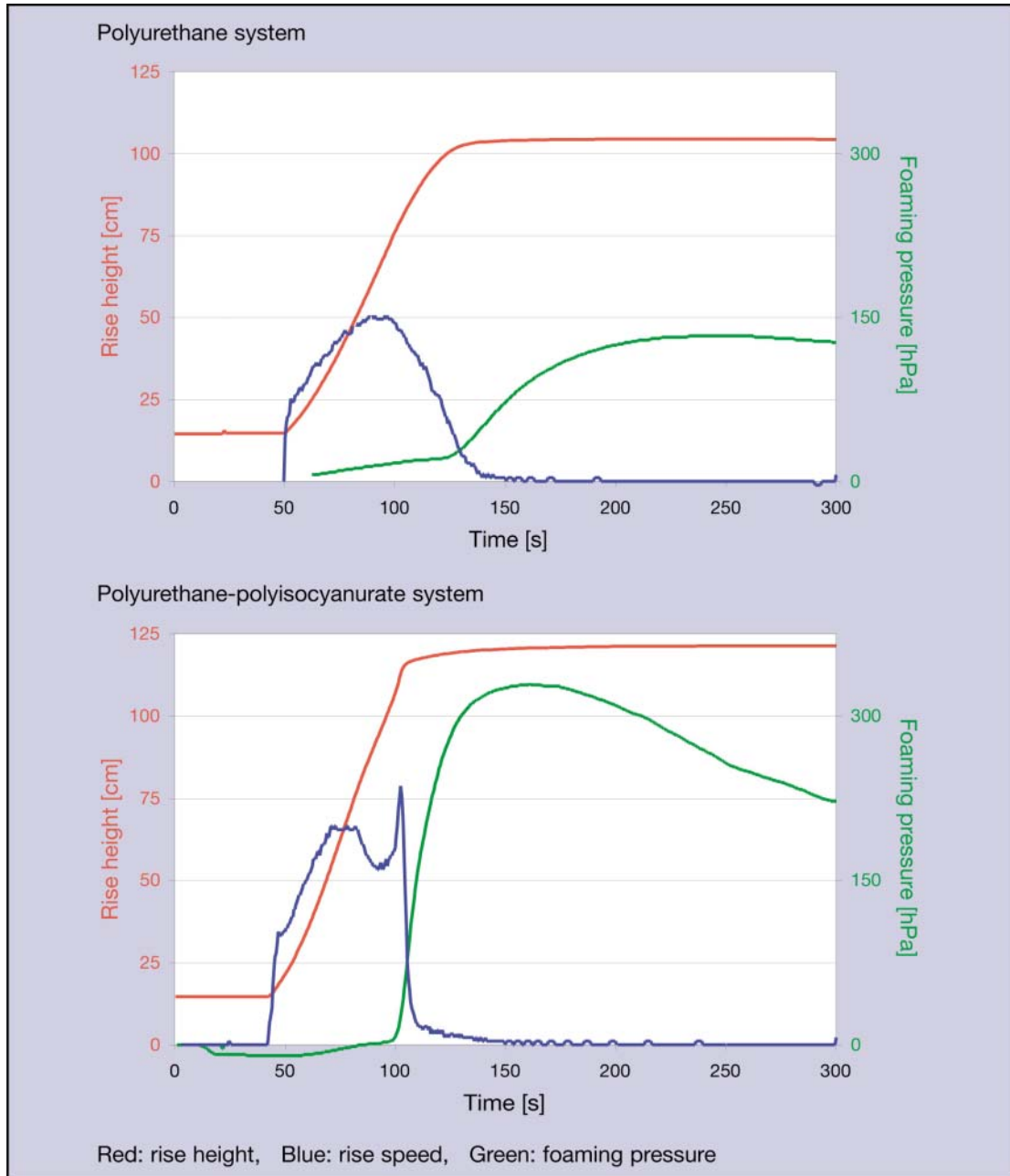


Fig. 11: Rise behavior of various foam systems

The rise speed of the foam depends on the composition of the reaction mix. The different effect of the activators used is particularly noticeable here. The following diagram shows the rise and rise speed of

the foam when activators are used which, to varying extents, accelerate the blowing reaction, i.e. the development of CO₂ and the formation of polyurethane.

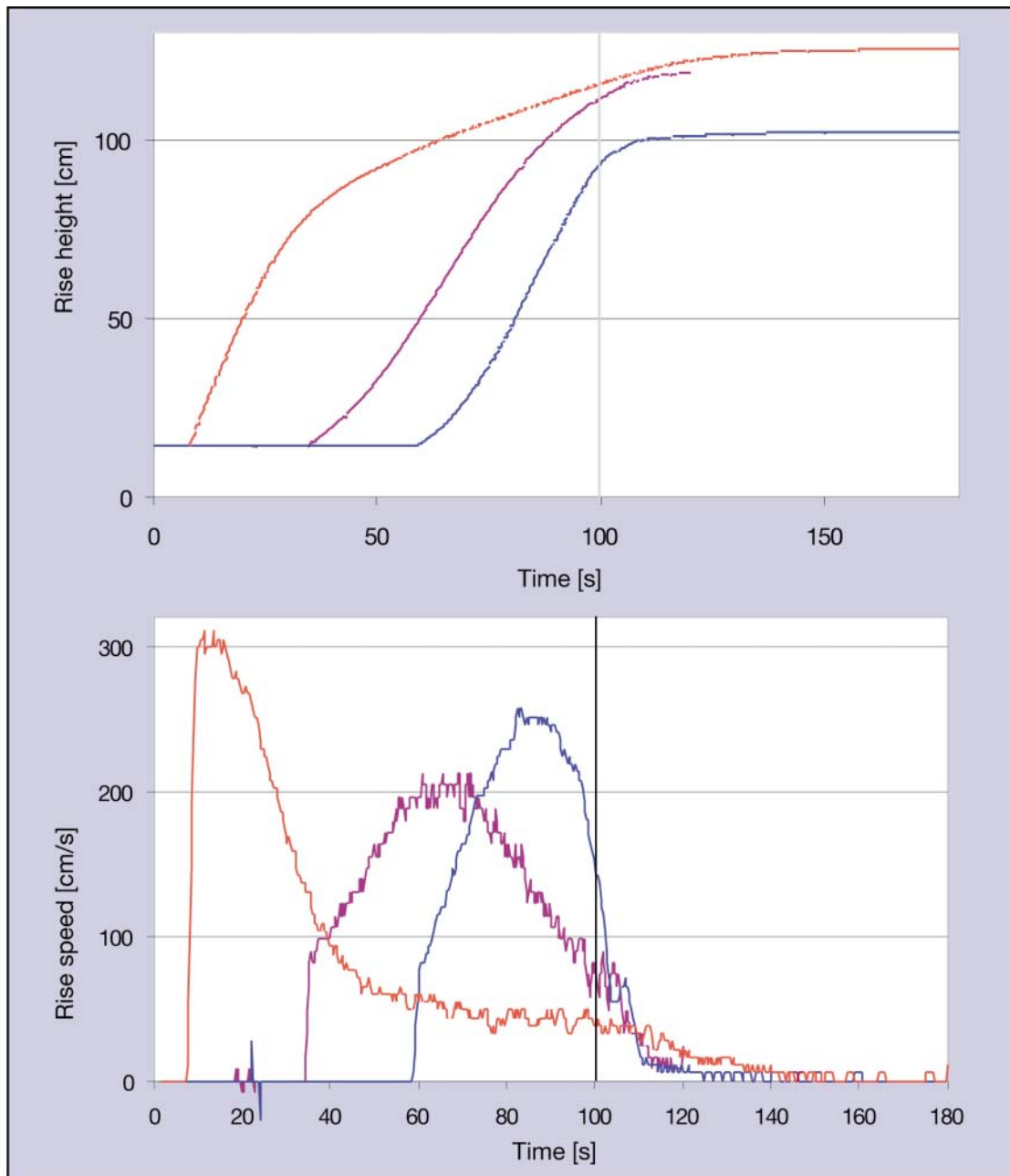


Fig. 12: Diverse rise behavior with different catalysts (activation adjusted to identical fiber time)

As can be seen, the start of foaming and the maximum rise speed are strongly affected by the various activators. The rise behavior (rise height) also changes. This method of influencing expansion and the course of the reaction is used when raw material systems are to be adapted for specific applications.

The increase in volume of the developing foam is also determined by the ambient air pressure. With

increasing air pressure the volume expansion is reduced, the reverse also being true. The changes in air pressure caused by the weather have a noticeable effect on foam density. With the same concentration of blowing agent in the reaction mix, foams of different density are obtained when foaming is carried out at various air pressures. The following densities were measured, depending on pressure, on a **free-rise foam** (foam can rise unhindered):

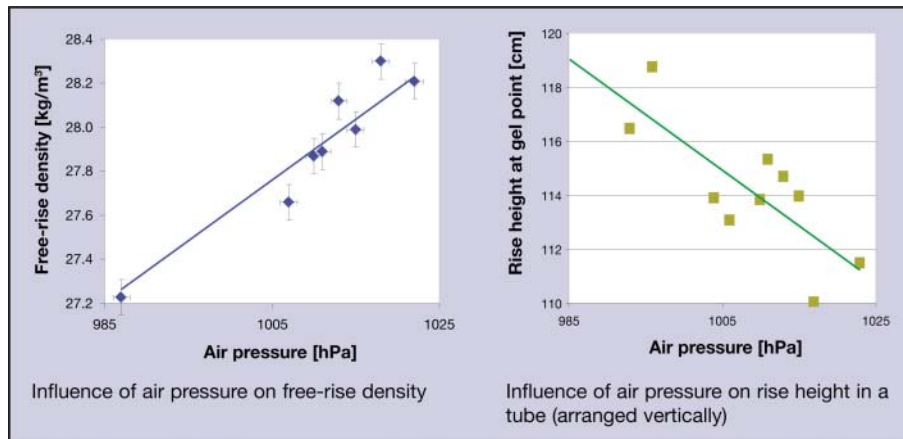


Fig. 13: Dependency of foam volume/foam density on air pressure

Different foam densities are therefore obtained when foaming at different altitudes.

When determining foam density it is therefore advisable to take the air pressure into consideration when producing the foam.

Rigid polyurethane foam is frequently produced in complex cavities between facings. Here, too, the foam has to rise against the ambient air pressure. An unusual increase in atmospheric pressure can prevent the cavity from being completely filled with foam by the predetermined amount of reaction mix. The change in foam level is shown in the following graph by filling a vertical tube with foam.

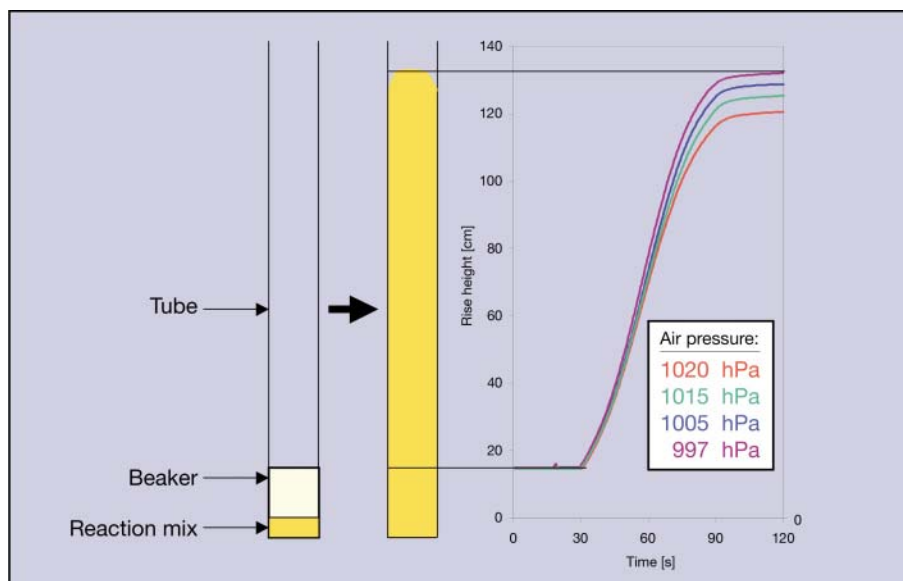


Fig. 14: Filling a vertical tube with foam

3.4 General rules for producing rigid polyurethane foams

To achieve optimum results when producing rigid polyurethane foam, certain parameters and regulations have to be adhered to. For applications in the building industry, rigid polyurethane foam is predominantly produced between facings arranged parallel to one another. The foam has to fill the cavity between two facings. A frame at the edges of the facings limits the spread of the foam in most cases. In its development phase the foam sticks to the facings, producing a more or less stable composite depending on the material of the facing and the surface quality.

The properties of the foam depend largely on the foam density (see Technical Information 14/2000 "Technical properties of polyurethane rigid foam as an insulating material in construction" [4]). The density accordingly has to be adjusted if certain properties are to be obtained. This is achieved by selection of the raw material system and by adjustment of the blowing agent content. In general, the density obtained with a free-rise foam cannot be achieved when filling a cavity with foam. The frictional resistance on the facings hinders the expansion of the

foaming reaction mix. This effect is increased by the counterpressure of the air that has to be expelled. As already mentioned, the foam has a flowing motion that brings about a cell orientation with corresponding impairment of the foam properties. Cell orientation can be reduced by "**overpacking**" the foam, i.e. more reaction mix is introduced than is necessary for filling the cavity. The amount of reaction mix should be measured in such a way that the foam fills the cavity before setting. For this reason the density of the free-rise foam is adjusted significantly lower than that of the foam to be produced. Overpacking of the foam results in considerable **foaming pressure** which has to be absorbed by a suitable device (for example a press or jig) until the foam is cured in order to obtain the desired dimensions. The pressure is dependent on the **degree of overpacking E**. This is defined by:

$$E = \frac{\text{required foam density}}{\text{free-rise foam density}}$$

The following table shows the dependency of the pressure on the degree of overpacking (valid for density range around 35 kg/m³).

Degree of overpacking E	Excess pressure (bar)
1.2	0.1–0.2
1.5	0.3–0.4
2.0	0.7–0.9
2.5	1.3–1.6

Table 2: Pressure as a function of degree of overpacking

The foam produced has to remain in the press or mold until only a slight, tolerable change in dimensions occurs on removal.

The **residence time** determines inter alia the cost-effectiveness of production. If the minimum residence time is not reached, the foam can bulge and

even split in the core region. The graph below shows the change in thickness of a foam sheet as a function of the mold residence time.

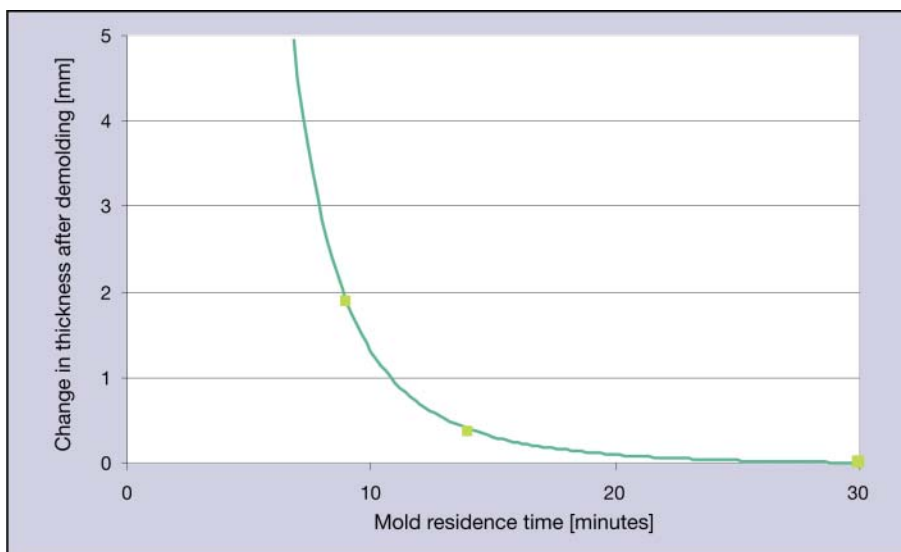


Fig. 15: Change in thickness of a foam board as a function of the mold residence time

Heat is evolved during the reactions leading to polyurethane, polyurea and polyisocyanurate. To keep the reaction rate at an adequately high level, only a little reaction heat should initially be carried away over the foam surface. Heat removal should be low even when applying the reaction mix to the facing or mold material. For this reason it is necessary to preheat the facing, the mold or the support device to 30 to 45 °C on the foam side.

During production of polyisocyanurate-polyurethane foams, the temperature should be considerably higher (up to 60 °C). However, at these relatively high temperatures losses of blowing agent can occur which become noticeable due to increased foam density and also due to bigger cavities/bubbles in the foam.

The heat loss over the facings and support device should also be kept within limits in the curing phase when the foam has filled up the predetermined space. Otherwise the foam can become brittle on the surface and adhesion to the facing may be inadequate.

The reaction mix should be introduced into the cavity in such a way that the rising foam spreads smoothly without over-rolling and completely expels the air. Vent holes should be arranged in such a way that the foam only reaches them when the air has been expelled. Air entrapments are avoided in this way. It is also recommended to bring the mold or support device into a position in which the flow direction of the rising foam is predetermined (for

example by tilting). Introduction of liquid reaction mix into already foaming reaction mix should be avoided by continuous mixing of the reaction components (for example by machine) and introducing the reaction mix into a cavity.

4. Process for producing rigid polyurethane foam

4.1 Hand mixing method

Mixing of the raw materials with the aid of a stirring rod was originally referred to as the hand mixing method. Nowadays this term is also used when the components placed in a mixing vessel are mixed with an electrically driven stirrer.

The hand mixing method is used mainly for developing and checking raw material systems on a laboratory scale and producing smaller rigid foam buns.

4.1.1 Production of test foams in the laboratory

Foam buns with a volume of 5 to 10 l are usually produced in the laboratory to determine or adjust the reaction times of a raw material system and the foam density. As the results have to be reproducible, the same production procedure and the same production conditions must be adhered to constantly.

The following equipment is recommended for production:

- 1 Pendraulik laboratory mixer, type LM-34 with foot operated switch (Pendraulik, Maschinen- und Apparate GmbH, D-31832 Springe, Fax: +49 (541) 50 29)
- 1 Plastic container with rinsing liquid for cleaning the mixer bowl
- 1 Lenart high-speed stirrer (Maschinenfabrik Paul Vollrath, Max-Planck-Str. 13, D-50354 Hürth, Phone: +49 (2233) 7 98 90)

Cardboard beakers, spirally wound with flanged sheet metal bases, 140/137 x 96 x 1 [mm] (Brüggen & Söhne, Postfach 5 23 03, D-52399 Düren)

- 1 Stopwatch (measuring accuracy 1 second)
- 2 Thermometers with 0.1 °C calibration
- 1 Balance (0.1 g scale)

Wooden rods approximately 200 x 2 [mm]

Molds for control pours (folded from packaging paper, base area 20 cm x 20 cm, height 14 cm)

- 1 Ribbon saw (for cutting test pieces) with table extractor fan Dust filter for the worker during sawing

Suitable temperature control container for the reaction component in the cardboard beaker (for example Dewar container with dry ice)

- 1 Barometer for measuring the air pressure

The temperature of the raw materials used should be above 23 °C. They can be brought to the required temperature (for example 20 °C or 23 °C) by simple cooling (see below).

In accordance with the foaming formulation, the polyol and the additives are weighed into a cardboard beaker and thoroughly mixed together by stirring. It is important that no raw material should adhere to the stirrer, so it is advisable to lift the running mixer from the mixture to spin off possible residues of raw material.

During mixing the mixer speed should **gradually** be increased to prevent the mix splattering. The

amount of blowing agent lost through evaporation must now be established by reweighing. The loss has to be replaced.

Approximately 1.5 times the amount of polyisocyanate mentioned in the formulation is then weighed out in a separate cardboard beaker.

The contents of the cardboard beaker are then adjusted to one of the above-mentioned required temperatures ± 0.5 °C. The raw materials have to be cooled due to the higher starting temperature. This is done by frequent brief immersion in the temperature control container while simultaneously stirring with a thermometer.

The amount of polyisocyanate which is to be added according to the formulation is now swiftly weighed into the cardboard beaker with the polyol mixture from the second cardboard beaker. The stirrer is switched on, immersed into the liquid at slow running speed and adjusted to the required speed (for example 1,000 rpm). The stopwatch is started at the same time as the stirrer is immersed. The stir time is usually 10 to 15 s. The reaction mix is poured into a packaging paper mold immediately after mixing and the cream time, fiber time, rise time and tack-free time are determined.

The control pour should be placed on a heat insulating material, for example a foam sheet, so the temperature of the reaction mix does not change during pouring and not too much heat is removed from the rising foam in the base region.

Cubes with an edge length of 10.0 cm can be sawn out of the core region of the foam bun to determine the density. This should not be done until after the foam has cooled.

4.1.2 Rigid polyurethane foam slabstock production by the hand mixing method

Slabstock is used in the form of sheets and other cut-to-size pieces, for example half shells of pipes. A great variety of composite elements can be produced from cut sheets by sticking on facings.

"Hand foaming" is the simplest method of producing small foam buns. It allows even highly viscous raw materials to be processed and solids and pastes (for example pigment pastes) to be simply mixed in. Moreover, only low investment is required. The disadvantages of hand foaming are the intensive

labor and the loss of raw material (residue remaining in the mixing vessel).

The following equipment is needed for a batch of about 50 kg:

- Mixing vessels with a capacity of about 100 l
- A high-speed stirrer which allows reciprocating and orbital movements (in simple-cases a hand drill with a stirring device is adequate)
- A balance with a measuring range up to 100 kg
- A balance with a measuring range of 1 to 2 kg and an accuracy of 1 g or a measuring cylinder for small amounts
- Protective goggles and gloves
- An extractor
- Cleaning agent

The total amount M_A of the batch is derived from the volume V of the mold to be filled with foam, the desired average density RD , the amount M_V of reaction mix remaining in the mixing vessel and the buoyancy difference A between the liquid reaction mix and the finished foam (= weight of the expelled air, approximately 1.2 kg/m^3 at 20°C) and the loss M_G of blowing agent resulting from the mixing and rise of the reaction mix. Therefore

$$M_A = V \times RD + V \times A + M_V + M_G \quad \text{Equitation 5}$$

The amount M_k of the components is calculated as follows:

$$M_k = \frac{\text{Total amount } M_A}{\Sigma \text{ of the parts by weight as per formulation}}$$

parts by weight of component as per formulation

An example of this is given below:

Volume of the molding $V = 1.2 \text{ m}^3$

Desired average density $RD = 35 \text{ kg/m}^3$

Buoyancy difference $A = 1.2 \text{ kg/m}^3$

Amount remaining in the mixing vessel $M_V = 2 \text{ kg}$
(determined in trials)

Loss of blowing agent $M_G = 1 \text{ kg}$
(determined in trials)

Equation 5 gives the batch amount as:

$$\begin{aligned} M_A &= 1.2 \text{ m}^3 \cdot 35 \text{ kg/m}^3 + 1.2 \text{ m}^3 \cdot 1.2 \text{ kg/m}^3 + \\ &\quad 2 \text{ kg} + 1 \text{ kg} \\ &= 42 \text{ kg} + 1.44 \text{ kg} + 2 \text{ kg} + 1 \text{ kg} \\ &= \mathbf{46.44 \text{ kg}} \end{aligned}$$

Formulation:

100	parts by weight of polyol
20	parts by weight of blowing agent
2	parts by weight of activator
<u>120</u>	<u>parts by weight of polyisocyanate</u>
242	parts by weight in total

The individual amounts are therefore:

$$\text{Baymer-polyol } M_{\text{polyol}} = \frac{46.44}{242} \times 100 = 19.190 \text{ kg}$$

$$\text{Blowing agent } M_{\text{TM}} = \frac{46.44}{242} \times 20 = 3.838 \text{ kg}$$

$$\text{Activator } M_{\text{Desmorapid}} = \frac{46.44}{242} \times 2 = 0.384 \text{ kg}$$

$$\text{Polyisocyanate } M_{\text{Desmodur}} = \frac{46.44}{242} \times 120 = 23.023 \text{ kg}$$

In the simplest case the mold in which the foam develops and finishes reacting can be a wooden box. Its interior faces should be smooth to facilitate removal of the foam bun. Mold release is facilitated either by paper (for example polyethylene-coated kraft paper or soda kraft paper) which has been laid or stuck in the mold before foaming or by release agents (for example release waxes). In the latter case, it is advisable to line the mold walls with sheet metal (for example aluminum sheet) or melamine resin-coated sheets. Suppliers of release agents include:

Acmos Chemie GmbH & Co.
 Industriestr. 37 + 49
 D-28199 Bremen
 Tel.: (+49) 4 21 51 89-0
 Fax: (+49) 4 21 51 14 15

IGEFA GmbH
 Europaallee 68-72
 D-50226 Frechen
 Tel.: (+49) 22 34 9 57 21-0
 Fax: (+49) 22 34 5 21 37

Goldschmidt AG
 Goldschmidtstr. 100
 D-45127 Essen
 Tel.: (+49) 201 173-01
 Fax: (+49) 201 173-3000

Buns are produced as follows:

The mold is set up and a release agent is applied to the inside walls. The mixing vessel is balanced on the scales, polyol, blowing agent and activator are poured in and mixed. The sequence of pouring is: large amounts before small and low-viscosity products before high-viscosity products. Finally, the polyisocyanate component is weighed in or a weighed-out amount is poured in. The components have to be thoroughly mixed with each other and poured into the mold within the cream time which is sometimes very short (approximately 15 to 40 s).

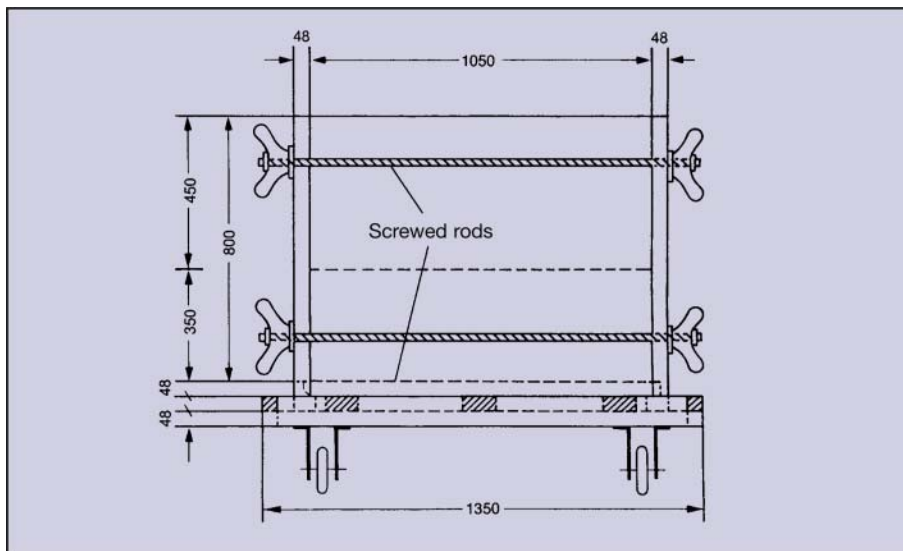


Fig. 16: End view of a box mold

Foaming in open molds is simplest. The reaction mix is poured into an open wooden box where it expands to form a foam bun. Although the pressure exerted on the side walls is relatively low, it must be taken into consideration in the design of the mold in view of the large areas involved. With polyurethane slabstock, a pressure of approximately 0.1 bar was measured at a density of approximately 35 kg/m³ and approximately 0.15 bar at a density of 60 kg/m³. Polyisocyanurate foams develop a significantly higher pressure.

Pressure build-up can be countered by opening the mold as early as 5 to 15 minutes after foaming the

bun and allowing the foam to expand freely. For this purpose two adjacent walls of the mold are released. The bun which is now expanding beyond the dimensions of the mold should be allowed to slide unhindered over the edge of the base. If the mold is not opened after these short time periods, there should be a wait of several hours to avoid pronounced bulging and even tearing of the bun. The bun should be demolded when the pressure on the mold has become sufficiently low. The moment should be determined experimentally.

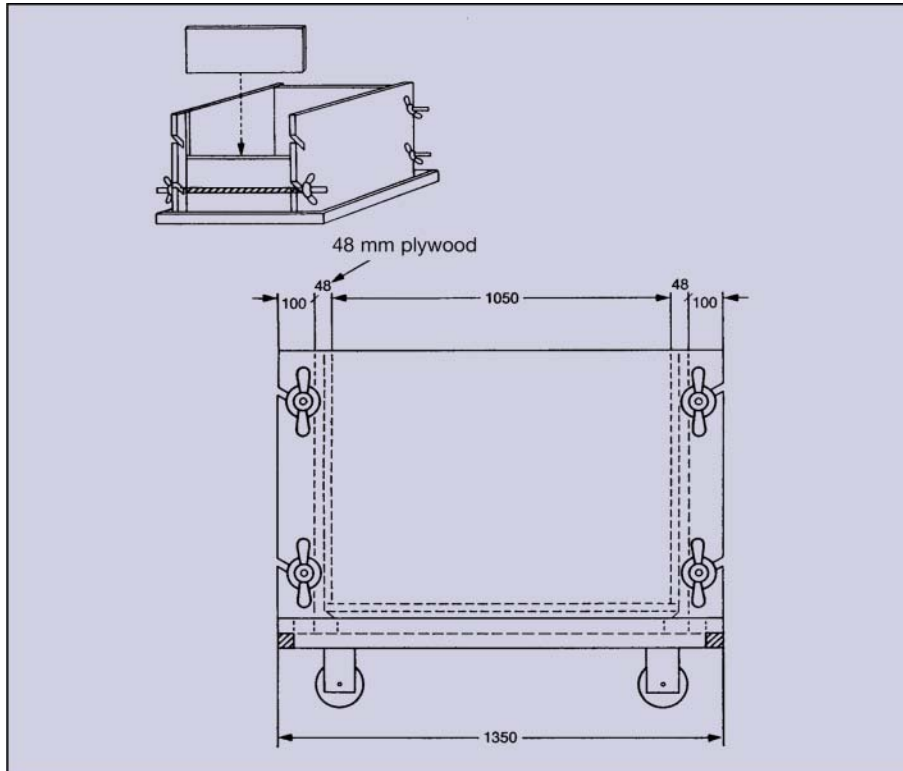


Fig. 17: Side view and perspective view of a box mold

The freely rising foam develops a crown and its cells are mainly oriented vertically (anisotropy). The development of a crown, which can cause a great deal of waste when cutting to size, can be reduced if the foam is allowed to rise against a **floating lid**. Once the reaction mix which has been poured in has been spread over the base of the mold and the expansion process has begun, a lid is placed on top of the reaction mix and is pushed up by the foam. The mold can be simply constructed in this case,

too, but should have smooth inner walls so the tightly fitting lid does not jam. The rising lid may have to be guided a little by hand. The normal weight per unit area of the lid is approximately 40 kg/m².

A completely rectangular bun can be obtained if the lid pushes against a stop and the foam is thus slightly compacted.

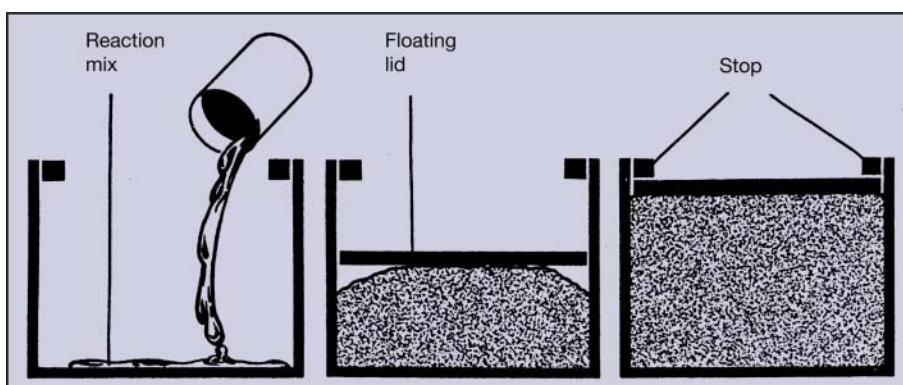


Fig. 18: Foaming against a floating lid

In some applications, the foam may only have a very slight cell orientation. This can be achieved by introducing the reaction mix into a mold which can be closed on all sides and compacting the foam more. The pressure exerted on the mold is correspondingly higher (see section 3: "Principles of rigid polyurethane foam production"). Long mold residence times have to be allowed for **foaming** under **pressure** as the buns could otherwise split.

Normally polyurethane raw materials are processed into foams with the aid of machines. Foaming plant for processing two or more components consists of at least one foaming machine and one molding device. The foaming machine can be seen as the heart of a plant.

It receives the liquid components, brings the components to a state in which they can be processed and keeps them there, meters them in the correct proportions, thoroughly mixes the components and dispenses the reaction mix. The following diagram shows the individual process stages.

4.2 Production of rigid polyurethane foam by machine

4.2.1 Foaming machines and foaming lines

The hand mixing process only plays a subordinate role in the production of rigid polyurethane foams.

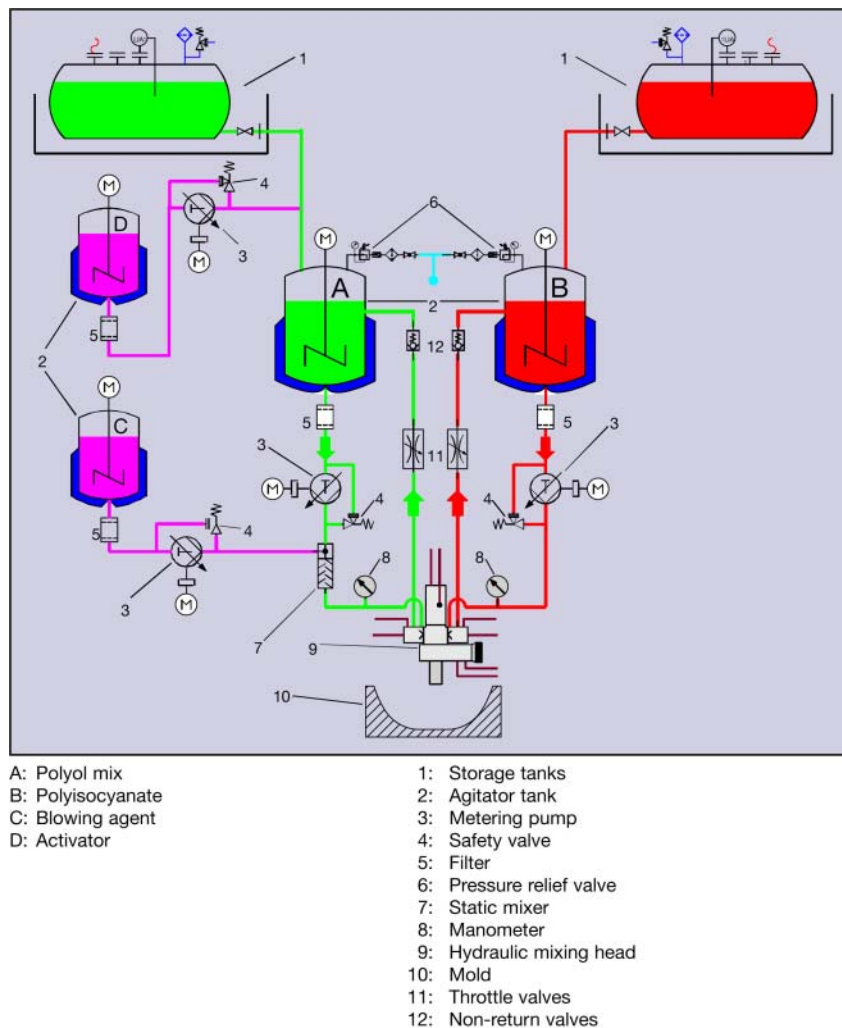


Fig. 19: Block diagram of a foaming plant

The two main components, polyol and polyisocyanate, are transferred from their storage containers into day tanks. In many cases, processing involves two-component systems, i.e. all the additives which are important for the reaction such as activators, stabilizers, blowing agents or flame retardants are already contained in the main components. However, individual mixing operations can also be carried out at **pre-mixing stations** or the additional components can be added directly into the metering lines of the pump units. The components are introduced into the day tanks in a processable state, i.e. temperature controlled and homogenized, and are kept in them. Dispensing units now convey the components in a set mixing ratio from the day tanks to the **mixing head**. The reactants meet here and are combined to form the reaction mix. This is discharged and then expands.

In general, a distinction is made between two machine systems: low-pressure and high-pressure

machines. The two systems are simple to define. On low-pressure machines the component streams at a pressure of 3 to 40 bar are conveyed to agitator mixing chambers, and on high-pressure machines the components are compressed to 150 to 300 bar by piston pumps and are then mixed by **impingement injection**. The high kinetic energy of the component streams as they enter the mixing chamber is used for mixing.

4.2.1.1 Low-pressure machines

With low-pressure machines, the components are usually circulated by gear pumps for a short time prior to pouring. Simple switch valves controlled by timing devices which determine pouring time ensure that the components enter the agitator mixing chamber simultaneously. After each shot, the mixing chamber has to be cleaned by aqueous flushing agents to remove any remaining reaction mix.

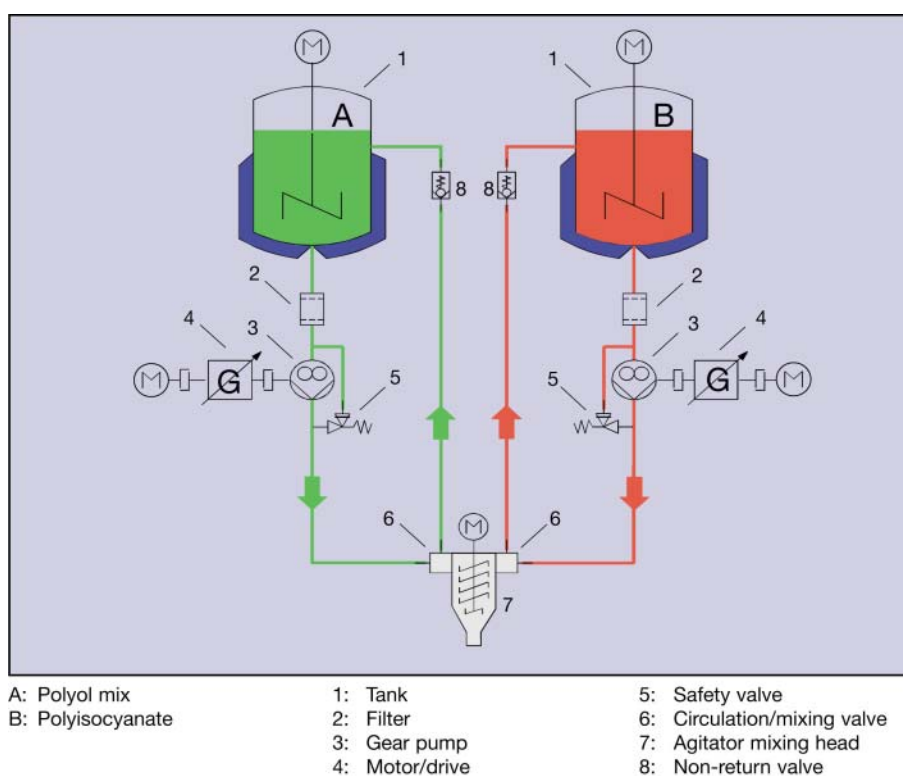


Fig. 20: Principle of a low-pressure machine

4.2.1.2 High-pressure machines

High-pressure machines can generally be divided into two categories: the in-line system and the recirculating system.

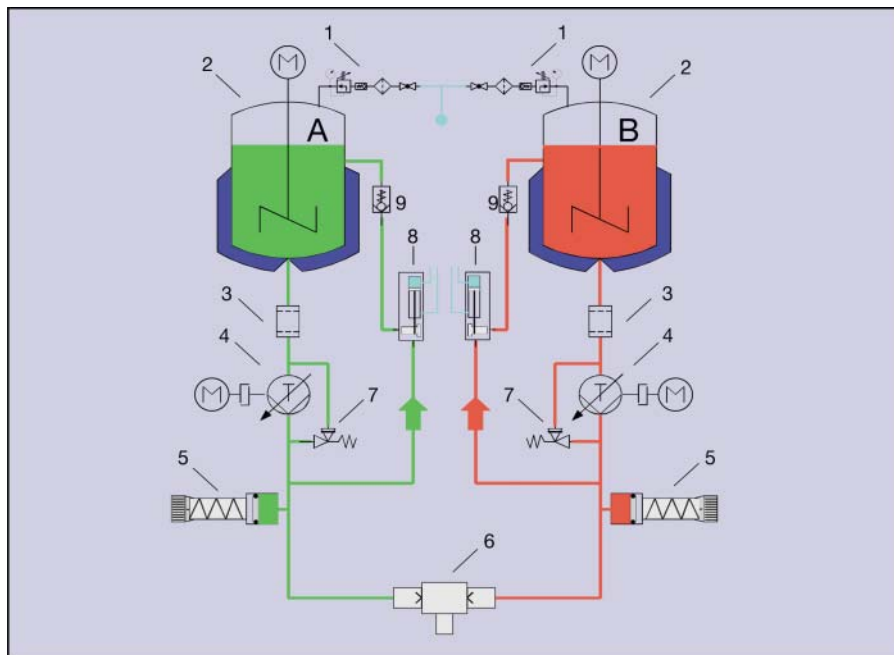
The in-line system

In the older in-line system, the components are fed directly from the day tank to the mixing head via metering units, i.e. at the start of pouring or when the metering pumps are started, the operating pressure builds up in the high-pressure lines to the mixing head. Once the adjustable initial resistance has been overcome, the injection nozzles in the mixing head open and the path is clear for the actual injection mixing.

The adjustability of the mixing chamber pressure necessary for high quality mixing is achieved by

twist discs downstream of the mixing chamber or by simple perforated discs. A *turbulence device* calms the emerging reaction mix into a laminar flow, separates the mix and guides it to the wall of the outlet pipe. At the end of each shot the small injection mixing chamber (approximately 0.5 to 2.5 cm³) is cleaned by a powerful air flush.

To synchronize opening of the spring-loaded nozzle elements, compensators which automatically adjust the volume of the pressure system and can thus delay the build-up of pressure are fitted in the high-pressure lines. At the end of the shot, coupled, individually adjustable relief valves instantaneously reduce the pressure on each side of the system to its original level. The nozzles close and the machine is ready for the next injection sequence.



- | | | |
|-------------------|--------------------------|---------------------|
| A: Polyol mix | 1: Pressure relief valve | 6: Mixing head |
| B: Polyisocyanate | 2: Tank | 7: Safety valve |
| | 3: Filter | 8: Relief valve |
| | 4: Metering pump | 9: Non-return valve |
| | 5: Compensators | |

Fig. 21: Principle of a high-pressure machine (in-line system)

The recirculating system

The general uncertainty in the case of control by means of resilient elements and the constantly growing demands on the end product have brought about further development in high-pressure technology.

The system in which the components are circulated from the day tank to the mixing head via the dispensing unit and back again has many advantages.

Firstly, the required throughput and injection pressure of the component streams are correctly adjusted before any material is poured, and secondly, switching from recirculating to injection mode takes place within positively controlled mixing heads (see below). All the operations responsible for perfect foam which is free of lead/lag problems are completed automatically within mechanically or hydraulically activated mixing heads.

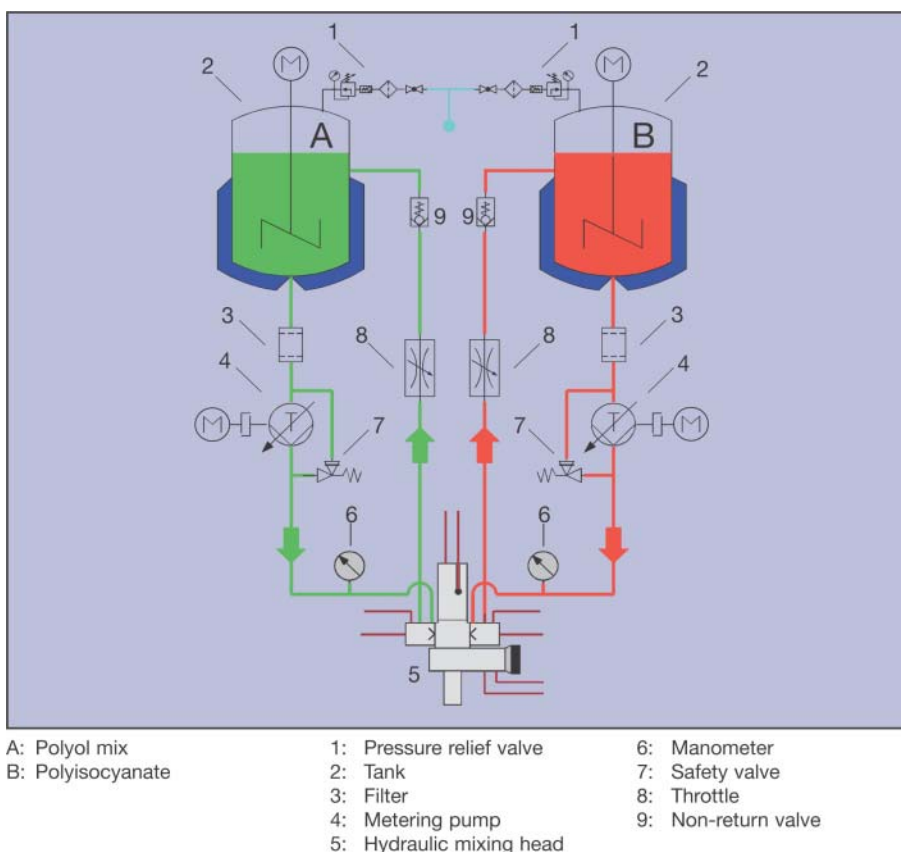


Fig. 22: Principle of a high-pressure machine (recirculation system)

4.2.1.3 Metering units and mixing heads

The components must be metered in the correct stoichiometric ratio, which must be reproducible with great accuracy, so precision pumps have to be used in low-pressure and high-pressure machines. Gear pumps of conventional design are usually used in low-pressure units. The output is controlled by varying the drive speed by means of infinitely variable drives and, more recently, by frequency converters.

Piston pumps (for example vertical, axial and radial piston pumps), which bring the components to the

required injection pressures of 100 to 300 bar, are used with high-pressure machines.

The most important part of a foaming machine is the mixing head, so there has been extensive R & D in mixing technology since rigid polyurethane foam production began.

The basic construction of agitator mixing heads and pressure-controlled mixing heads is based on the in-line system (low-pressure and high-pressure). There are, however, several different designs of self-cleaning positively controlled mixing heads.

Agitator mixing heads

The raw materials introduced into the upper part of the mixing chamber are intensively mixed by rotating variable-speed stirrers as they proceed to the outlet. There are various types of stirrers, for example pin-type, screw, paddle, straight arm paddle and worm stirrer.

Pressure-activated mixing heads

The two injection nozzles are directly opposite one another in a small cylindrical mixing chamber. The nozzles open when the component pressure overcomes the spring bias of the nozzle pins. Recently, nozzles with minimal pressure loss (Fig. 23) have been used which can be controlled by air via a membrane so the component pressure has to overcome the initial pneumatic resistance to open the nozzles.

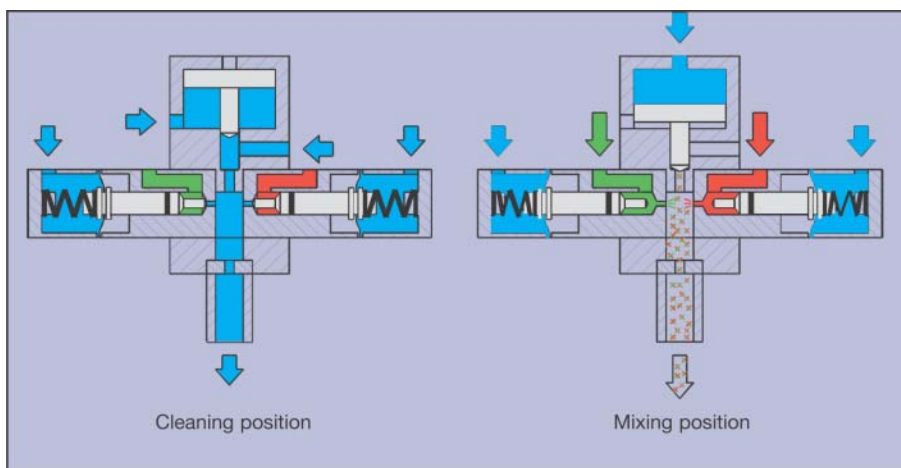


Fig. 23: Pressure-activated mixing head (nozzles with minimal pressure loss)

Hydraulically operated mixing heads

In these types of mixing heads, as well as actual injection mixing, the component streams are switched under high-pressure from circulation to injection mode. The nozzles are thus synchronously switched at the start and at the end of the injection process. Many mixing heads of this type continue to be self-cleaning in design, i.e. at the end of the shot the reaction mix remaining in the mixing chamber is expelled with the aid of cleaning pistons.

Details of the various types, for example piston-controlled mixing heads, needle valve nozzle, Hub-schieber and rotating mixing heads will not be given here.

Manufacturers and suppliers of foaming plant and mixing heads can provide this information:

Hennecke GmbH
 Polyurethane Technology
 Birlinghovener Str. 30
 53754 Sankt Augustin
 Tel.: (+49) 22 41/339-0
 Fax: (+49) 22 41/339-204
 E-Mail: hennecke@hennecke.com

Machine parts which come into contact with the reaction mix (mixing chamber, outlet pipe, perforated pour pipe etc.) have to be cleaned regularly. Suppliers of suitable solvents are:

Kraemer & Martin GmbH
 Zum Siegblick 37 - 45
 D-53757 Sankt Augustin
 Tel.: (+49) 22 41 54 97-0
 Fax: (+49) 22 41 6 41 77

Jäkle Chemie GmbH & Co. KG
 Matthiasstraße 10-12
 D-90431 Nürnberg
 Tel.: (+49) 911 326 46-0
 Fax: (+49) 911 326 46-6
 E-Mail: chemikalien@csc-jaekle.de
 Internet: www.csc-jaekle.de

4.3 Continuous production of slabstock foam

If very long, wide buns are to be produced, the discontinuous method will very soon be found to have limitations due to the fact that the reaction mix must be dispensed into the mold and spread within the cream time, so continuous production is the most economical method and the one which is preferred industrially.

The reaction mix is laid down on a conveyor belt by a foaming machine with a movable mixing head.

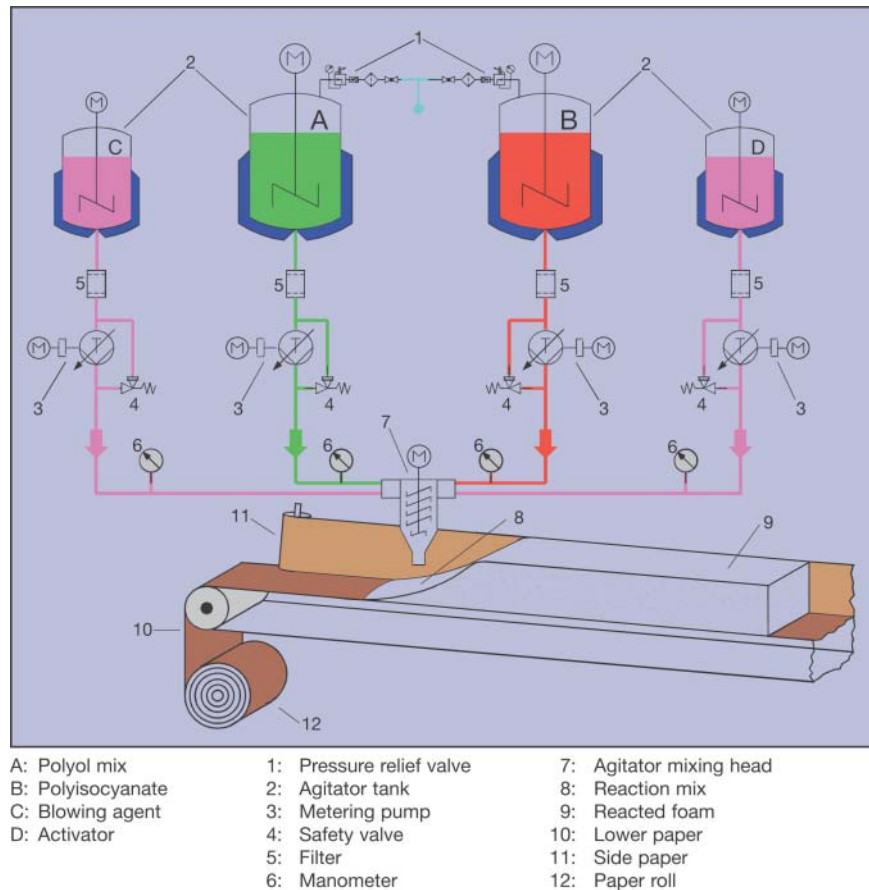


Fig. 24: Principle of a plant for continuous slabstock foam production

A length of paper folded into a U shape acts as a release agent and at the same time serves to transport the developing bun. The side walls also have to be designed as conveyor belts because of the direct lateral pressure occurring after the expansion process.

To avoid crown formation and reduce cutting losses, the flat-topped slab method is mainly used for continuous production. With this process a paper web runs in the rising foam zone and is pressed onto the foam with the aid of appropriate devices.

This results in a bun with an almost rectangular cross-section.

The quotient of the length of the press section and the speed of the conveyor belt is comparable to mold residence time in the discontinuous production process. Much shorter mold residence times are possible with continuous production than with discontinuous production. The short distances which the reaction mix has to cover mean that cream and fiber times can be shorter than with discontinuous production and curing is therefore quicker.

Bun widths of 1.00 to 1.50 m, bun heights of 40 to 100 cm, densities of 30 to 200 kg/m³, conveyor belt lengths of approximately 15 m and production speeds of 2 to 7 m/min are the norm.

With continuous production of slabstock, particular attention should be paid to the angle of slope φ which the foaming reaction mix can assume in relation to the horizontal. The output A, the width of the

bun B, the density RD, the height of the bun H and the speed VB should be coordinated in such a way that a stable state is attained. The expansion line must be stationary with respect to its surroundings. The belt inclination angle α can be increased to produce higher buns, but particular care must then be taken to avoid undercutting expanding reaction mix with liquid reaction mix.

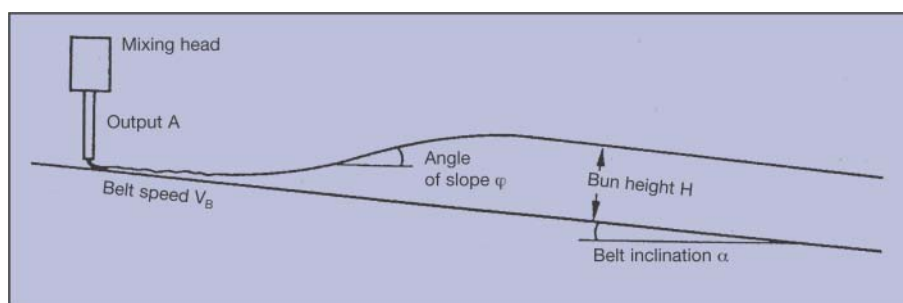


Fig. 25: Expansion line in continuous slabstock production

4.4 Production of rigid polyurethane foam boards and panels with facings (insulating boards, sandwich panels)

The production of boards and panels made of rigid polyurethane foam with a variety of facings is both economical and efficient.

If rigid facings are used on both sides, sandwich panels combining low weight, high rigidity and excellent insulating capacity are obtained in one operation (see Technical Information No. 9/1999, "Importance and usage of polyurethane metal faced panels in industrial buildings" [5]). When using diffusion-resistant facings, the optimum initial thermal conductivity of the rigid polyurethane foam is retained, i.e. metal sandwich panels with optimum heat insulating capacity are obtained.

There are numerous possibilities for combining rigid polyurethane foam with various facings. They range from paper-laminated insulating boards to high-grade, highly insulating cold store panels with a foam core up to 24 cm thick and torsion-resistant, profiled sheet metal facings.

In addition, there are numerous possibilities for combining rigid polyurethane foam with flexible

facings on one side and rigid facings on the other side. However, this asymmetric construction means that attention must be paid to possible warping.

A range of discontinuous and continuous methods has been developed for producing boards and building panels in the most technically and economically viable manner for the type of board or panel and the proposed capacity.

4.4.1 Continuous production of insulated rigid polyurethane foam boards and sandwich panels

Continuous laminators are used for the large scale production of rigid polyurethane foam slabs with facings. They consist of two continuous conveyor belts aligned one above the other and capable of absorbing a certain amount of pressure. The gap between the two belts is adjustable, allowing the thickness of the panels to be adjusted. The belts run parallel to one another, and the space between the reversal points is usually at least 10 m so there is a press section of corresponding length. The press section is usually equipped with circulating air heating.

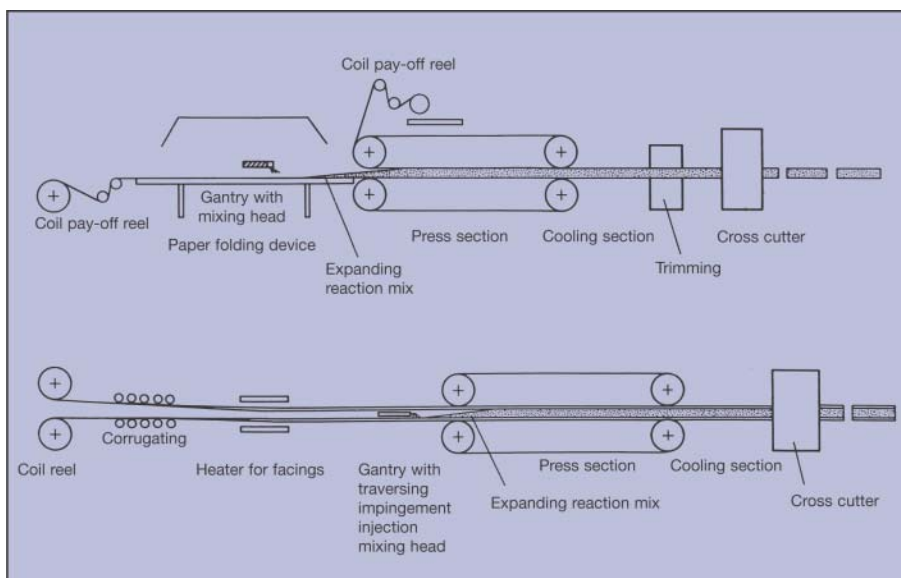


Fig. 26: Diagrammatic representation of a continuous laminator with flexible facings (top picture) and rigid facings (bottom picture)

The facings - papers, foils, mats and metal sheets - are wound off rolls and fed into the space between the conveyor belts. In rare cases, rigid facings, for example chipboard or plasterboard, are used. They are taken from a stack and fed continuously, with no gaps, into the continuous laminator. A support which also acts as a spacer is needed for the upper facing. Metal sheets usually pass through a corrugating station before they reach the conveyor belts.

In the region prior to the press zone, the reaction mix is applied to the inside face of the lower facing. It must be adjusted in such a way that it expands quickly and reaches the upper facing at the start of the press section. The developing board or sandwich panel is carried along by the conveyor belts as a result of the contact pressure.

A side wall prevents lateral escape of the foam. Different panel designs and operating modes require an appropriate side seal in each case. This forms the side wall of the continuous mold and generally takes the form of static plates which can be adjusted in width (for flexible facings) or an accompanying side sealing chain made of blocks (for profiled metal facings).

The production rate of the continuous laminator depends on the curing rate of the fresh foam and on the length of the press section. Nowadays there are raw material systems capable of providing 3 cm thick panels which are rigid enough for further

processing after a residence time of only 1 to 2 minutes in the press section. After leaving the belt, the panels are cut to the desired length.

Panels can be produced in thicknesses of approximately 30 to 240 mm with many different types of corrugation. Roof panels are usually more deeply corrugated than wall panels to ensure high rigidity.

The choice of facings depends on the intended application. Bituminized paper and soda kraft paper which has been dyed black is used for floor insulating boards. Polyethylene-coated soda kraft paper, polyethylene-coated glass mats and mineral fiber mats are used primarily for roof insulation boards. The required diffusion resistance is achieved with metal facings over 50 μm thick, for example aluminum foil. These facings can be coated, for example, with paper, polyethylene or a bonding agent for improved adhesion. The fire performance requirements of the insulating boards should be taken into account when choosing facings.

Corrugated coated steel or aluminum sheets are generally used to produce roofing and wall panels. Extra care is needed during distribution of the reaction mix due to the corrugations. The corrugated sheets can only be fed into the continuous laminator at a relatively narrow angle, a fact which imposes certain limitations on the height of the reaction mix dispenser.

The quality of the metal sheet surface and the coating has a decisive influence on the composite and the surface quality of the panel produced. Inadequate adhesion of the foam and defects in the foam structure can be caused by poor quality of the surface and coating (see Technical Information No. 7/1998 "Influence of steel facing backing paints on the quality of metal-faced polyurethane sandwich panels" [6]). The problems mentioned can often be largely eliminated by corona treatment of the foam side surface of the facings (Corona equipment supplier: Agrodyn Hochspannungstechnik GmbH, Bisamweg 10, D-33803 Steinhagen, Fax: +49 (5204) 92 10 33). Obviously, facings must be free of dust and grease.

Constant temperature control (heating) of the metal facings is very important and greatly influences the

outcome of foaming. The temperature should be 35 to 40 °C in the dispensing zone.

Dispensing the reaction mix

The reaction mix applied to the lower facing expands to as much as 40 times its thickness. Even distribution of the reaction mix is therefore essential, particularly in the case of thin, smooth laminates. There are various methods for this. Linear dispensing oscillating over the width of the laminate is the most widely established method because of its simplicity and versatility. With this method, a mixing head, of the impingement injection type, for example, is moved back and forth at right angles to the direction of travel of the belt, pouring or spraying the reaction mix onto the lower facing in a zig-zag line.

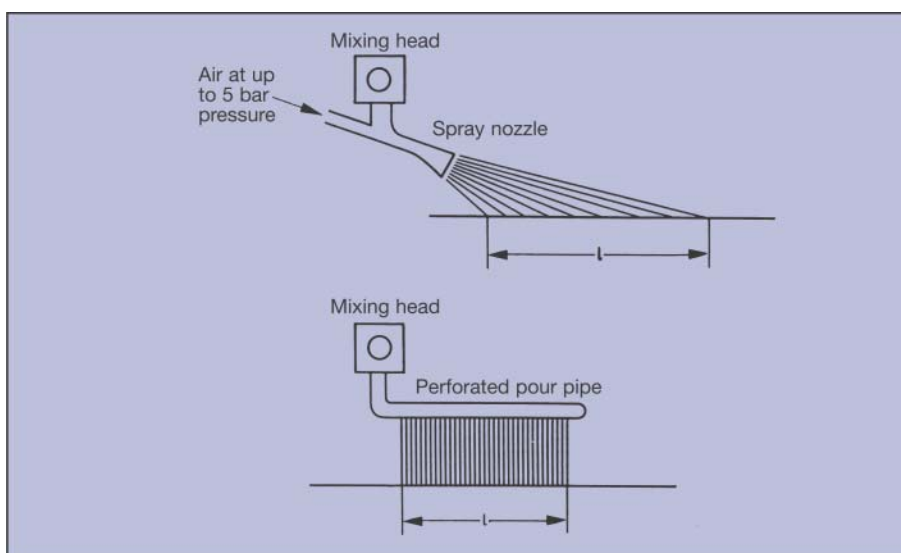


Fig. 27: Linear dispensing of the reaction mix by spraying or pouring

The speed V_m of the mixing head is approximately 50 to 150 traverses per minute. The length l of the spray jet or of the curtain of reaction mix dispensed from a tube with multiple perforations may be up to 30 cm. With the requirement for double overlapping and a belt width B , the maximum belt speed V_B is calculated from the equation:

$$V_B = \frac{l}{2B} \times V_m \quad \text{Equitation 6}$$

Assuming from Equation 6 that $V_m = 150$ m/min., $l = 0.3$ m and $B = 1$ m then the maximum belt speed is

$$V_{B \max.} = \frac{0.3 \times 150}{2 \times 1} = 22.5 \text{ m/min.}$$

Belt speeds are normally between 2 and 12 m/min depending on panel thickness and the press section. In certain cases, 20 m/min can be achieved.

Oscillating dispensing of the reaction mix is not possible at a higher belt speed. Stationary mixing heads and, if need be, special dispensing methods are used in such cases.

Foam rise profile

Viewed from the side, the reaction mix forms a clearly visible foam rise profile.

The stable state necessary for good, reproducible foam quality is achieved when the foam rise profile

remains stationary with respect to its surroundings. To create these conditions the speed of the moving belt must be coordinated with the reaction cycle.

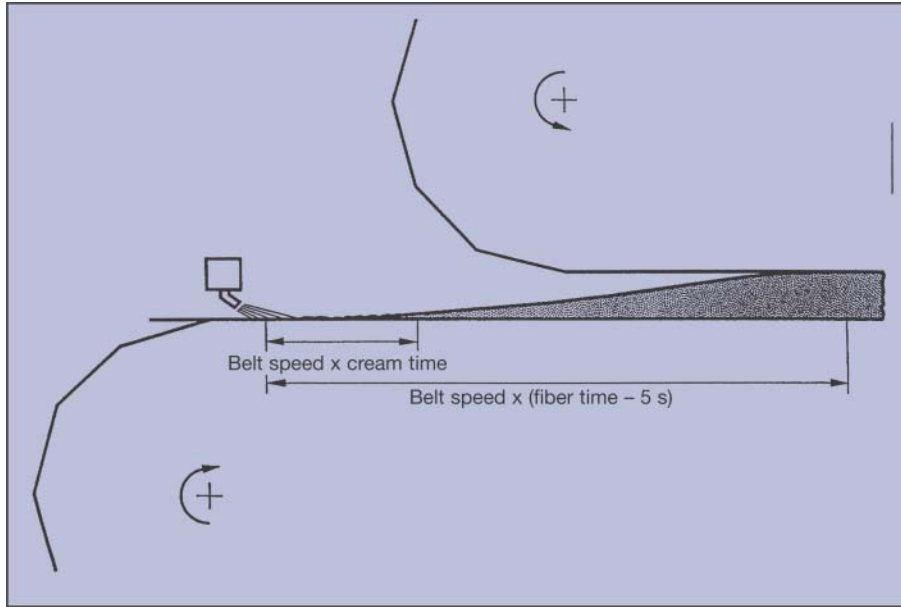


Fig. 28: Diagrammatic representation of the foam rise profile of the reaction mix on a continuous laminator

The output of the foaming machine and the width and height of the laminate are fixed. In theory, the belt speed should be adjusted so the foaming mate-

rial contacts the upper facing without exerting pressure.

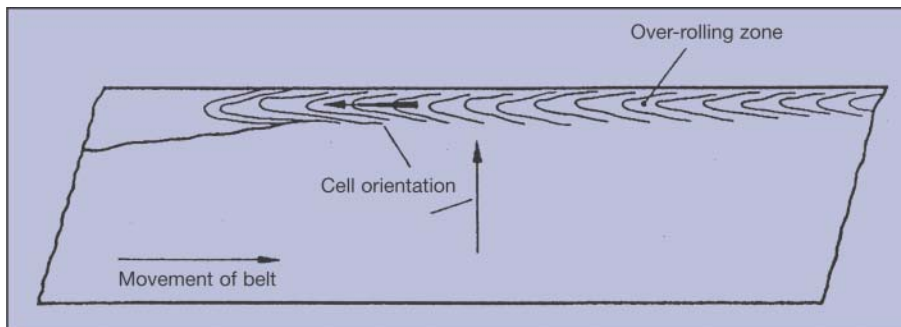


Fig. 29: Diagrammatic representation of "over-rolling"

In practice this adjustment is not possible. The space between the facings must be completely filled with foam. A certain pressure is necessary for this process and must be absorbed by the reaction mix without the mix being forced back against the direction of movement of the belt. Any such forcing would result in "**over-rolling**". Slight over-rolling

leads to smoothing of the slightly wavy surface of the rising reaction mix, leaving no bubbles or air entrapments under the upper facing. Pronounced over-rolling has considerable disadvantages as the rising reaction mix is disrupted by the resultant friction. The cells are stretched in the direction of travel of the belt and this adversely affects compressive

strength and tensile strength perpendicular to the surface of the laminate. Vertical tears can also occur in the laminate.

During production, therefore, it is important to observe the foam rise profile into the laminating tunnel, and lamps and optical devices can be used for this purpose.

The **contact time**, i.e. the time needed by the foaming reaction mix to reach the upper facing after dispensing, is determined by putting a small cube of foam onto the lower facing before dispensing the mix. The stopwatch is switched on as the foam cube is wetted with reaction mix. The contact time is obtained when the cube touches the upper facing. It should be 3 to 5 s shorter than the fiber time.

With the aid of temperature control devices, the foam rise profile and thus the production speed, but also the density distribution, can be influenced. The temperature of the raw materials, the lay-down zone, the facings and the conveyor belts should remain constant to maintain a stable state.

The raw materials should normally be kept at a temperature of 20 °C, with the dispensing zone heated to between 25 and 30 °C, and belt temperatures between 35 and 45 °C. If metal sandwich panels are being produced, the metal facings should be preheated to have a temperature of 35 to 40 °C in the lay-down zone.

Curing zone

The pressure needed to give the expanding reaction mix the desired thickness or shape is in most cases very low, because the mix should not have

started to cure before contacting the upper belt. Nevertheless, the laminating tunnel should be designed to withstand an internal excess pressure of 0.5 bar when insulating boards or panels in the typical 30 to 50 kg/m³ density range are being produced. This internal pressure is not purely hydrostatic but is the difference between the forces of expansion, for example gas pressure or thermal expansion, and those forces which hold the foam together. The strength of the latter increases continuously during the curing phase and subsequent cooling until the difference ultimately becomes insignificant and the foam can safely leave the press section of the laminator.

Finally, as with foaming in molds, it is the dimensional stability of the finished article which determines mold residence time - in this case in a moving mold. Due to the short flow distances which the reaction mix has to cover, reactivity can be very high. With panels 3 cm thick, residence times as short as 1 minute are possible.

Cooling zone

After leaving the press section, the boards/panels are sawn to the desired length. Heat accumulation is to be avoided during subsequent stacking as it can cause wavy surfaces, bulges, foam tears and scorching. If these features do appear, the boards/panels have to be cooled before stacking. To save space, this can take place in a device with gaps between the boards/panels so that air passing in-between acts as a coolant (for example a cooling rack). The residence time should be determined empirically.

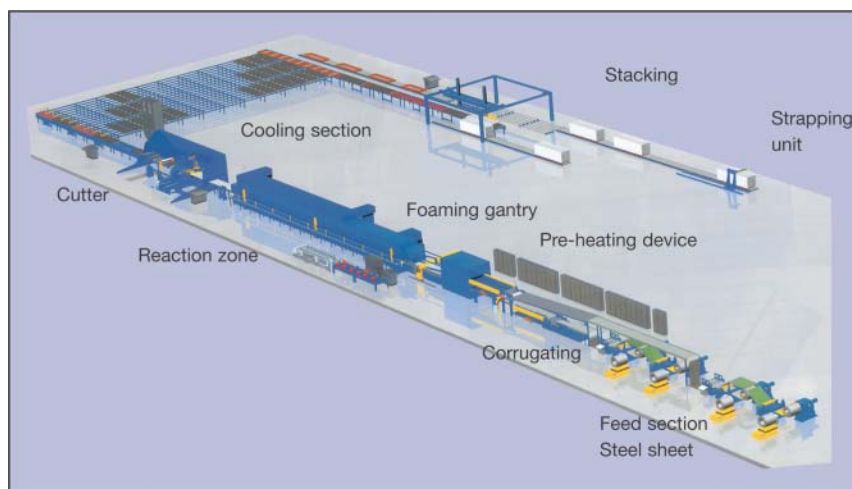


Fig. 30: Hennecke's Contimat plant (continuous laminator)

4.4.2 Discontinuous production of sandwich panels

While continuous production of sandwich panels by means of a continuous laminator is particularly suitable for mass production, discontinuous production is preferred for small orders, very thick panels and when it is also necessary to include molding frames and fixings in the foam.

Simple hinged molds or devices which can absorb greater pressures, such as presses (including multi-daylight presses) are used here as jigs. Manufacturers and suppliers of jigs/presses of this type include:

Hennecke GmbH
Polyurethan Technology
Birlinghovener Str. 30
53754 Sankt Augustin (Birlinghoven)
Tel.: (+49) 22 41 3 39-0
Fax: (+49) 22 41 3 39-2 04
E-Mail: hennecke@hennecke.com
www.hennecke.com

Dieffenbacher GmbH & Co.
Heilbronner Straße
75031 Eppingen
Tel.: (+49) 72 62 65-0
Fax: (+49) 72 62 65-2 97
E-Mail: dse@dieffenbacher.de
www.dieffenbacher.de

Makron Oy
Norokatu 5
FIN-15201 Lahti
Tel.: (+3 58) 3 81 23 12
Fax: (+3 58) 3 7 33 12 99
www.makron.fi

Manni S.p.A.
Via Campione, 11
S. Biagio di Bagnolo S. Vito (MN)
Italien
Tel.: (+39) 03 76 41 50 41
Fax: (+39) 03 76 41 52 64
E-Mail: info@mannipresse.it
www.mannipresse.it

Siempelkamp GmbH & Co
Siempelkampstr. 75
47803 Krefeld
Tel.: (+49) 21 51 92-30
Fax: (+49) 21 51 92-5360
E-Mail: info@siempelkamp.com
www.siempelkamp.com

The facings and, where necessary, the frame should be placed horizontally in heatable jigs

(presses). If the frame is not part of the panel but part of the jig (for lateral absorption of the foaming pressure), it must be treated with a release agent, for example wax-based, if the foam comes into direct contact with it.

The facings should not be fixed when the jig is shut, so that they can expand freely when subjected to heat expansion. This can be achieved, for example, by a ledge in the molding frame. Sagging of the horizontal upper facing should also be reduced by the use of spacers (for example cubes of foam). Stresses in the facings which could cause bubbles and wavy panel surfaces can be avoided by these measures. However, spacers can impede undisturbed foaming of the reaction mix, so their position and number should be determined carefully. It is better to fix the upper facing to the support device (press) by vacuum.

The reaction mix is introduced into the cavity between the facings and frame, where it expands and fills the cavity by displacing the air. Section 3 ("Principles of rigid polyurethane foam production") describes points which require special attention.

The reaction mix is usually introduced through the molding frame. This has to be provided with inlet and venting holes. The inlet holes (diameter corresponding to the inlet pipe) and the venting holes (2 to 5 mm in diameter) are made horizontally or vertically in the frame according to the dispensing position. The inlet openings should be located above the anticipated liquid level of the reaction mix.

In rare cases, the reaction mix is poured, with the mold open, onto the lower facing, which is fixed in a frame. The support device is then closed, and the upper facing which is fixed to the "cover" of the jig by vacuum, is applied simultaneously.

The seal between the frame and the facings has to be made carefully, taking care to prevent reaction mix from escaping at the seams. Specially chamfered facings or corrugations which are pressed against one another by the foaming pressure, making it impossible for liquid or foam to escape, are widely used.

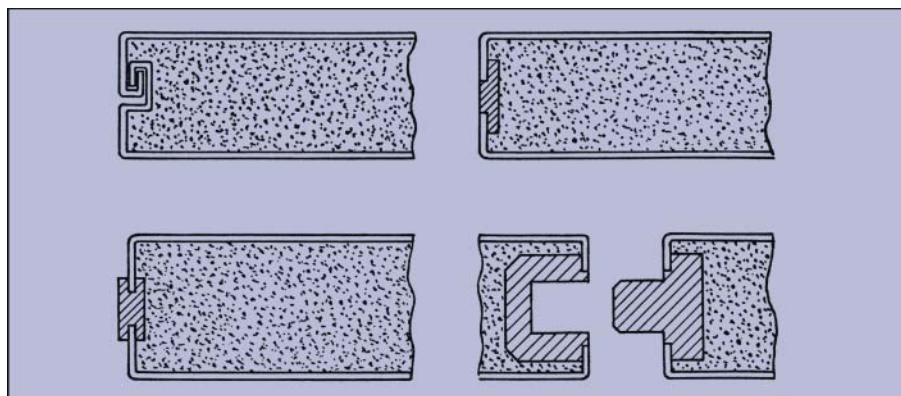


Fig. 31: Examples of panel designs and connections

The escape of foam at the venting holes can lead to sharply reduced foam quality, with cell orientation and considerable entrapments of gas. The venting holes can be backed by gas-permeable fabric or flexible foam.

Long flow paths should be avoided as they impair the quality of the expanding foam, and relatively high densities are required to achieve adequate compaction. Short flow paths can be achieved by

- using a perforated pour pipe
- prior distribution of the reaction mix
- optimization of the filling position.

Entrapment of air in the expanding reaction mix can be avoided by the use of a suitable filling method and position and by correct location of the venting holes.

Various filling methods and mold positions are possible for the production of sandwich panels:

Single-shot method

The reaction mix can be introduced in one shot through one filling hole. (With very long, narrow panels, it may be an advantage to locate the filling hole at one end.)

Multiple-shot method

When producing sandwich panels over 6 m long, the reaction mix is frequently introduced in several shots via inlet holes on the long side. Owing to the long overall shot time, the reaction mix from the first shot will inevitably have foamed to a considerable extent, while the reaction mix from the last shot is just starting to foam. This causes foam defects due to over-rolling, bubbles under the facings, lack of dimensional stability of the foam etc. The following diagram is a plan view of the foaming process (horizontal foaming position) shortly before filling the cavity by the three-shot method.

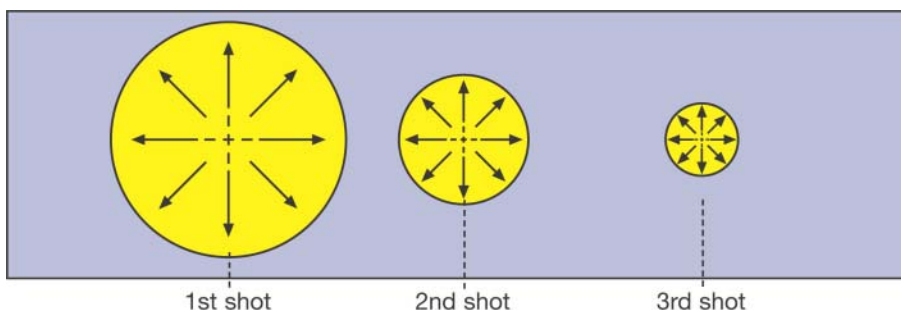


Fig. 32: Schematic view of the distribution of the reaction mix with the multiple-shot method after the third shot (40 s after the first shot)

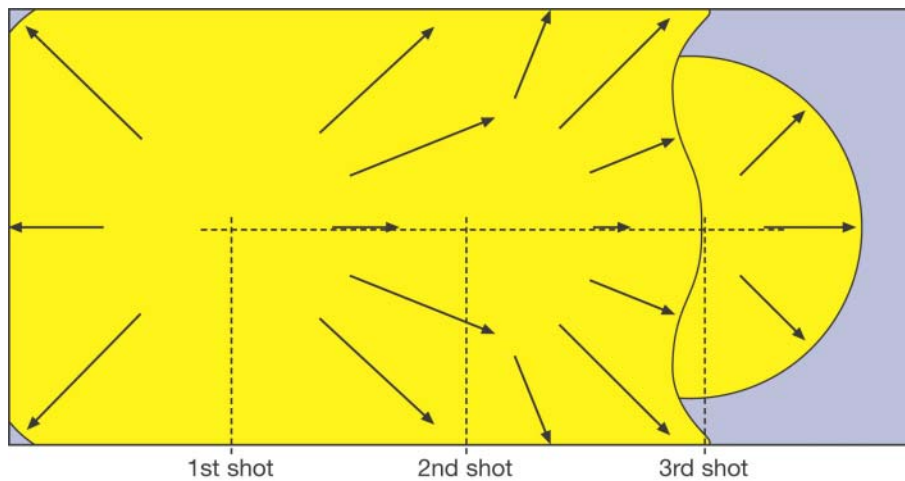


Fig. 33: Schematic diagram of over-rolling in the expanding reaction mix during the multiple-shot method (80 s after the first shot)

Problems such as this can be avoided by dividing the cavity into several compartments by means of partitions and filling these individually. The **partitions** can consist, for example, of rigid foam strips which should be firmly anchored in place, otherwise they could be displaced from their position as the foam pressure builds up. A disadvantage of this process is that the partitions show through on the facings.

The disadvantages of the multiple-shot method can be avoided by using the **VarioCast** process developed by Bayer AG with equipment from Hennecke GmbH. This process can also be used with the lance, withdrawal and perforated pour pipe methods.

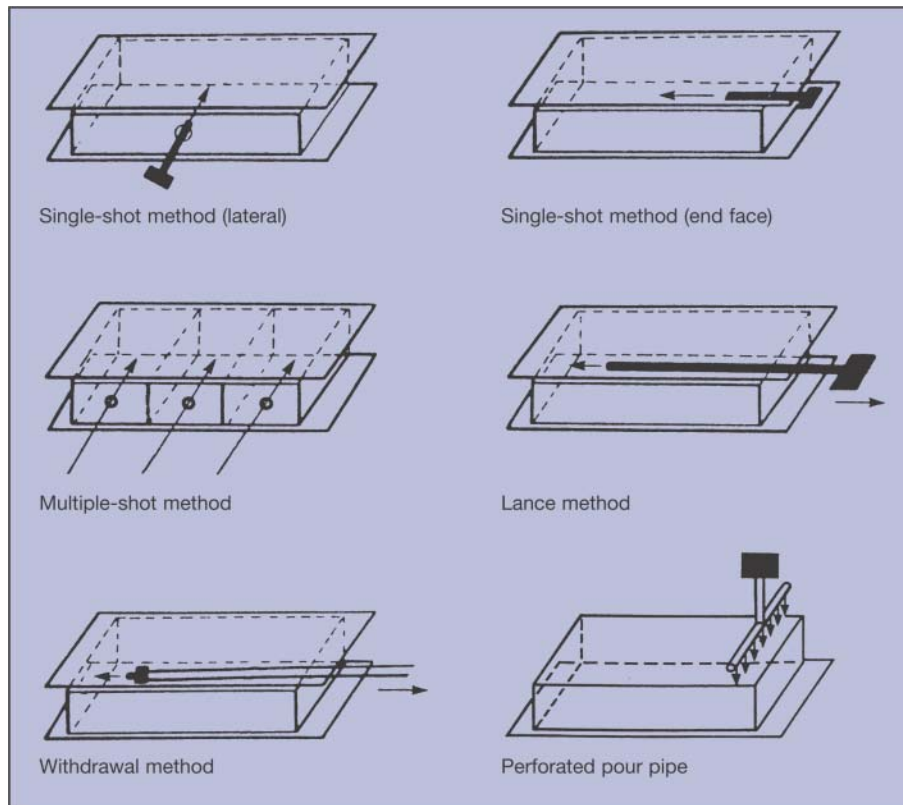


Fig. 34: Dispensing methods for the discontinuous production of sandwich panels with a rigid polyurethane foam core

Lance method

A perforated pour pipe which is two-thirds the length of the mold is attached to the mixing head. The inlet hole is located at one end, and the lance is slowly withdrawn from the mold during filling.

Withdrawal method

This is similar to the lance method, except that the entire mixing head is initially situated inside the mold and is gradually withdrawn as filling proceeds. A suitably small mixing head is required here.

Multiple-outlet pouring

The reaction mix is dispensed from a perforated pour pipe which is moved over the open mold. A press with a hinged lid which can be closed immediately after dispensing, for example, is needed for this method. Multiple-outlet pour pipes are used in the **Panel Foamer** plant supplied by Hennecke GmbH.

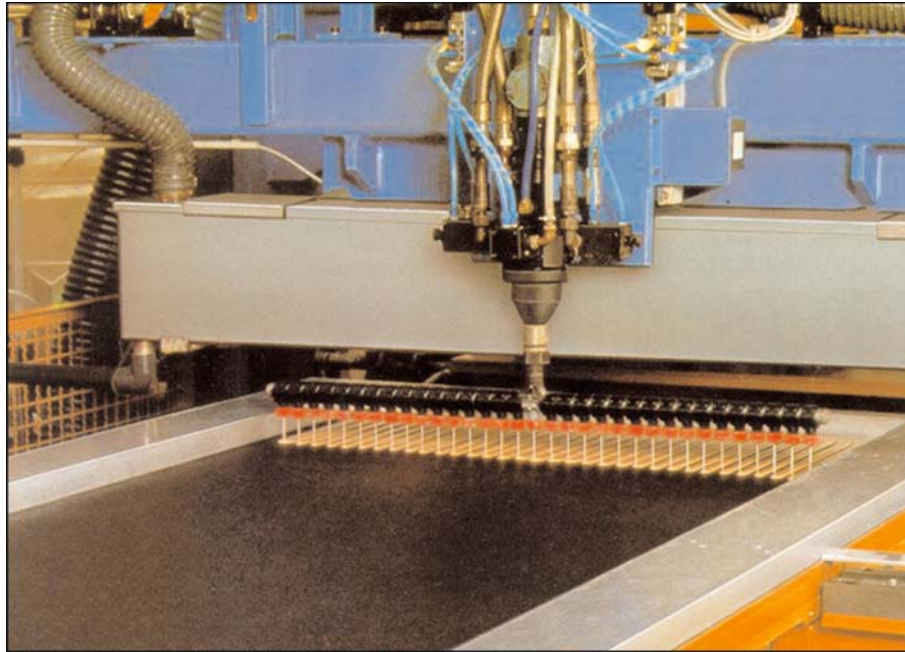


Fig. 35: Panel Foamer

Mold positions

Horizontal: suitable for the single-shot, multiple-shot, lance and withdrawal methods and for the perforated pour pipe

Vertical: as above, but not suitable for the perforated pour pipe

Tilted: generally used only with the single-shot and multiple-shot methods

The jigs and their locking devices must be designed to withstand any tendency to deform under expansion pressure.

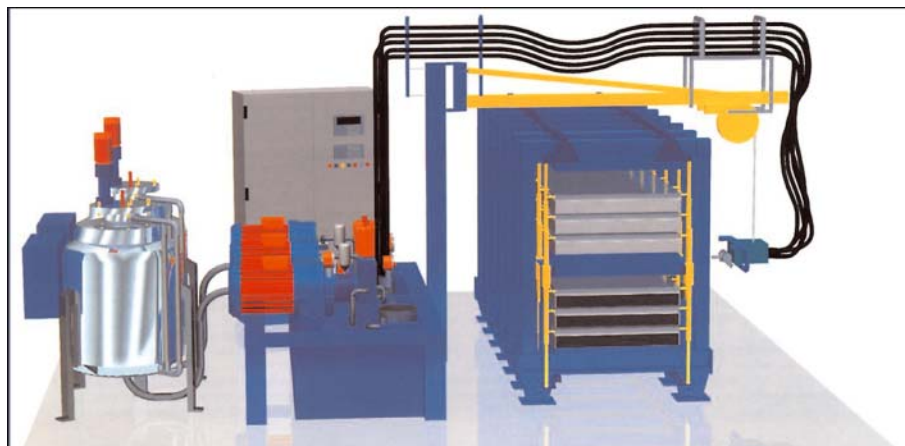


Fig. 36: Discontinuous production line (multi-daylight press) for sandwich panels

4.5 In-situ rigid polyurethane foam

In-situ (spray-in-place) rigid polyurethane foam is produced directly on site by pouring (usually layer-by-layer) and by spraying and is therefore subject to many influences. When foaming flat roofs, walls of buildings and storage tanks, the inevitable variations in temperature and atmospheric humidity have to be taken into account. The advantage of in-situ foam is that it is not necessary to transport large volumes of foam, just the raw materials and the foaming machine. Even complicated surfaces can be coated with a relatively uniform foam layer by this method.

4.5.1 Spraying

The reaction mix consisting of the two components, polyol (with all the additives including blowing

agent) and polyisocyanate, is best produced by high-pressure machines and sprayed without an air supply. The machines are transportable and equipped with long delivery hoses to the mixing head. The hoses are sheathed in a heat-retaining device, and the mixing head is integrated into a gun so spraying can be started and stopped as desired.

The reaction mix is adjusted for quick reaction and curing of the foam by the addition of appropriate catalysts. On reaching the surface, the mix foams spontaneously and forms a heat insulating layer. Its reactivity can be adjusted in such a way that the mix does not run off vertical or overhead faces. The level of operator skill required is about the same as when applying paint.



Fig. 37: Thermal insulation and sealing in one operation

The dependency of the foaming process on temperature has been referred to on many occasions. When foam is applied by the spray method, particular attention needs to be paid to the temperature of the raw materials and of the surfaces being sprayed. The reaction mix very quickly loses heat on cold surfaces because the layer sprayed on is so thin (approx. 1 mm). As a result, the foam tends to run off inclined surfaces, its cell structure alters and it becomes brittle. Unfortunately, these problems cannot always be solved by heating the reaction mix or by increasing the amount of catalyst, because if the cream time is too short, the reaction mix will react in the air and be unable to spread satisfactorily. This will result in a more granular surface texture. Ideally, the temperature of the surface being sprayed should be above 15 °C, although this depends on its thermal capacity and thermal conductivity. The surface must also be dry, otherwise the polyisocyanate will react with any water present, leading to a coarse cell structure and poor adhesion. Adhesion will also be impaired if the surface is oily or dusty. Metal surfaces should be treated with an anticorrosion agent. When spraying out of doors, the relative humidity of the air must not be too high (<70 %), and steps must be taken to prevent any of the reaction mix being blown by the wind onto surfaces which are not to be sprayed. Tents or wind shields erected around the spraying area help to overcome this problem. It must, however, be stressed that spraying leads to greater quantities of both PMDI vapor and reaction mix fog. This means that efficient ventilation systems and breathing masks (preferably fresh-air respirators, because filters can become blocked by the foaming reaction mix) are essential.

Spraying generally yields layers of foam about 1.5 cm thick with a density of between 35 and 70 kg/m³. Thicker layers can be produced by repeated spraying, but the density of the topmost layers of foam will be lower since they are being applied to a warmer substrate. Due to heat dissipation, a layer of higher density, known as the "foam skin", forms on exposed foam surfaces. When several layers of spray foam are applied, it is important to ensure that this foam skin adheres perfectly to the next layer.

Suppliers of spraying machines:

Gusmer Europe S.A.
Sector Industrial Masia d'en Barreres
Rambla Torre de l'Oncllet, 7
Apartado Correos no 203
08800 Vilanova i la Geltrú, Barcelona
Tel.: (+34-93) 8 11 53 00
Fax: (+34-93) 8 93 96 00
E-Mail: info@gusmer-europe.com
www.gusmer-europe.com

Isotherm AG
Industriestraße 6
CH-3661 Uetendorf
Tel.: (+41-33) 3 46 02 02
Fax: (+41-33) 3 46 02 09
E-Mail: isotherm@isotherm.ch
www.isotherm.ch

4.5.2 Layer-by-layer application

Layer-by-layer application is a favorite method of producing in-situ foam. It is used when cavities cannot be filled with foam by a single filling process, because performance of the foaming machine is limited and the foam pressure cannot be absorbed.

With the layer-by-layer method, the reaction mix is poured in layers into the cavity, which is open at the top. Tight shuttering absorbs the low foam pressure, eliminating the need for an additional jig.

After several foam layers have been produced, the shuttering can be moved upwards. At the same time the sheet metal sheathing which is often used (for example for raw material tanks) can be built up for the foam.

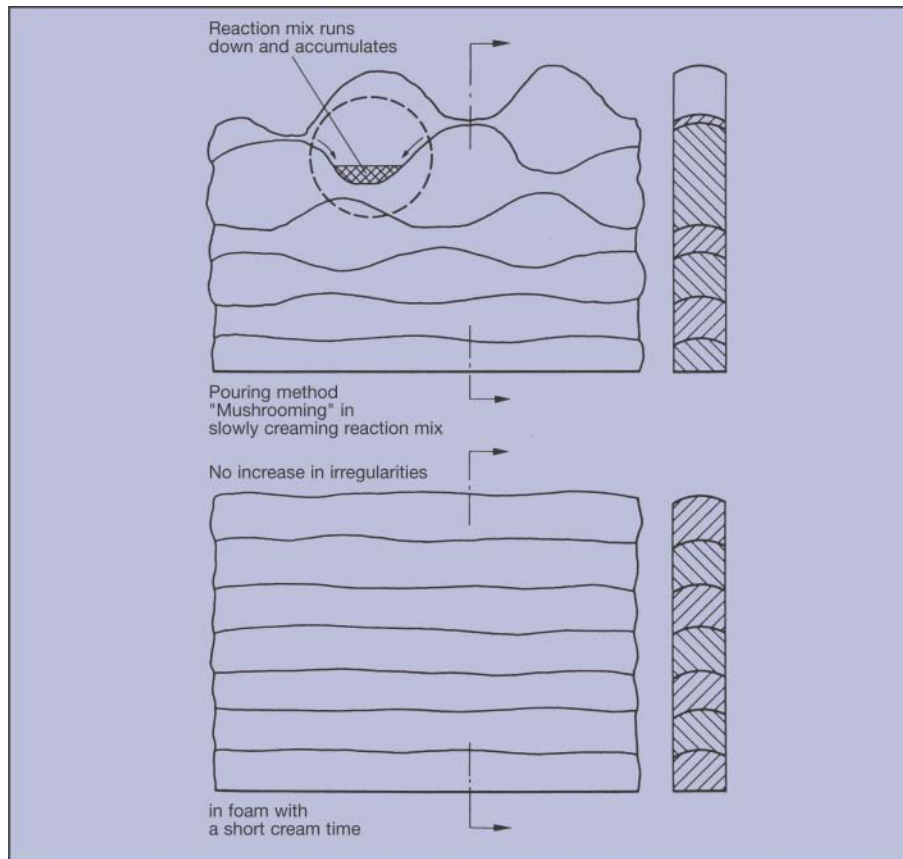


Fig. 38: Layer-by-layer application by the pouring method

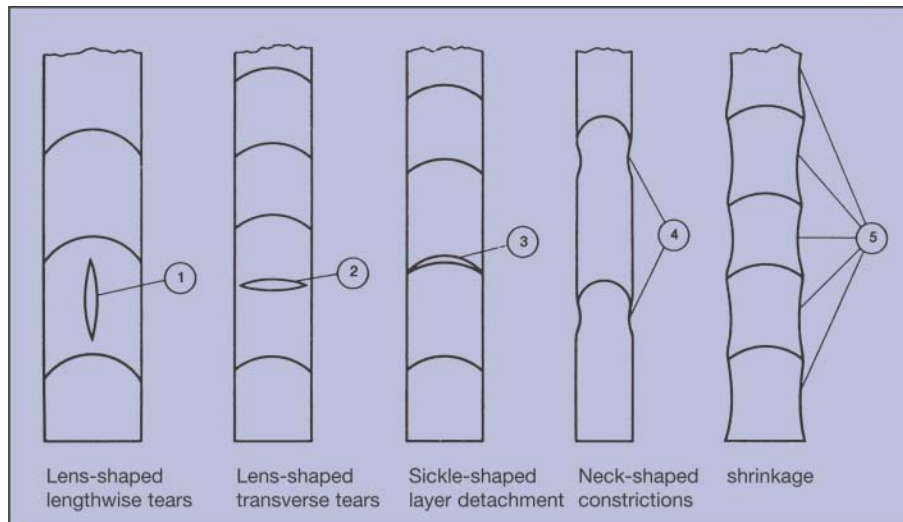
As already stated in the section on spraying, the surface temperatures should not fall below 15 °C in the layer-by-layer method either. In any case pre-treatment with an anticorrosion paint is advisable for objects made of metal. Moisture should be excluded during insulating work. It is important to introduce the reaction mix uniformly. Even slight irregularities in the first layer can adversely affect the following layers. Reaction mix which has been applied uniformly to an irregular layer flows into the valleys before the start of foaming and rises from here to an extremely high foam layer which can result in cavities. This undesirable increase in irregularities can be largely offset by a short cream time. The creamy consistency of the reaction mix prevents it from flowing away and collecting in the valleys.

The height of the individual layers should be limited to about 20 cm. The rigidity of the foam and its

dimensional stability could otherwise be impaired. The waiting time before applying the subsequent layer is between 10 and 30 minutes. Longer waiting times impair adhesion of the layers. Layering of mechanically shaped foam (which has been leveled, for example, by sawing or planing) can impair layer bonding (venting of the cut cells).

Sheathing made of galvanized sheet steel has to be pre-treated for better adhesion to the foam. To this end, the metal sheet should be treated with phosphoric acid-containing wash primer and an appropriate paint applied.

As a rule, the insulating thickness is limited to 150 mm by the output of the foaming machine, but also by the difficulties described in producing even layers.



	Cause	Remedy
①	insulation too thick, activation too high, lead or lag included	thinner insulation, possible composite insulation, lower activation, better control of foaming machine
②	layers too thick, activation too high, lead or lag included	thinner layers, lower activation, better control of foaming machine
③	layers too thick, lower layer machined, time for application of next layer too short or too long, activation too high, lead included	thinner layers, do not machine foam, keep to waiting time of 10 to 30 minutes, lower activation, better control of foaming machine
④	layers too thick with small cross-section, density too low	thinner layers increase density
⑤	raw material and ambient temperature too low, incorrect formulation activation too low	correct raw material and ambient temperature check polyol mix and machine adjustment, optimize mixing, higher activation

Fig. 39: Defects with layer-by-layer application

4.5.3 One-component foam

Polyurethane one-component foam is a blowing agent-containing, moisture-curing resin-catalyst mix excellently suited to assembly, insulating, filling, bonding and sealing.

The resin-catalyst mix is based on a pre-adduct (prepolymer), produced from isocyanate and a polyol formulation and containing additives (cata-

lysts, stabilizers, flame retardants) necessary for foaming. Foaming takes place as a result of spontaneous evaporation of the blowing agent dissolved or emulsified in the prepolymer. The foam cures as a result of the free NCO groups of the prepolymer reacting with the ambient humidity.

One-component foams have been used in the building industry since the beginning of the 1970s, and various types of foam are available for diverse

applications. Most are "fixing" and "filling" foams which are used to fix door and window frames and shutter boxes, and for filling cavities and openings in walls to provide sound and heat insulation.

5. Quality control, safety

5.1 Control of production conditions and the end product

There are various ways in which the manufacturer of polyurethane and polyisocyanurate foams can achieve and maintain the required level of quality. An essential requirement here is a monitoring system to detect fluctuations or faults in good time. A quality management system in accordance with ISO 9000 and corresponding process and operating instructions can help to avoid defects and to discover them.

As already stated, the temperature of the raw materials considerably influences the progress of the reaction. Storing the raw materials in temperature-controlled rooms or containers ensures that polyisocyanate and polyol are always processed at the same temperature (a temperature of approximately 20 °C has proved to be ideal in most cases).

There should be a regular check on whether the correct formulation is being used. On foaming machines, the output of the pumps for the individual components should be monitored. Although new plant is usually equipped with flow meters, the test curves should be checked. It is also important for the components to arrive in the mixing head simultaneously and in the correct amounts for the formulation. To this end, a strip of reaction mix is poured onto a paper web. When the foam has cured, color differences will clearly show whether one raw material component is reaching the mixing head too soon when the machine is switched on (lead) or whether one raw material component continues to flow longer than the other when the machine is switched off (lag), which can occur, especially with pressure-controlled mixing heads.

The mixing quality is monitored by cutting the strip of foam lengthwise and crosswise and examining it for striations.

Once the pumps have been correctly adjusted, lead or lag problems eliminated and the mix is satisfactory, a control pour is made (see Section 3). Cream time, fiber time, rise time, tack-free time and possi-

bly gas release time are determined and compared with the predetermined values.

The control pour is cut open and the cell size and cell structure are assessed.

When the foam has cooled, a cube with 10 cm sides is cut from the control shot. The free-rise density is determined by weighing.

These tests should be carried out before foam production begins (i.e. at the start of each shift). It is also a good idea to make a control pour after any lengthy period of down time, to check for lead/lag, cell structure, cell size and possibly density. Temperatures and, if necessary, working pressures should also be regularly checked.

The mold to be filled should be inspected to check that the locking devices are functioning correctly, and for tightness, possibly release agent application, cleanliness and temperature.

The operation of the pumps should be monitored when the reaction mix is introduced in order to ensure that the required amount is dispensed into the mold. The foaming process can often be observed directly, for example in block foaming in open molds or continuous laminating. With closed molds, the emergence of foam from the vent holes indicates how the mold filling process is progressing.

After demolding, a check is made to see whether the mold has been completely filled. In the case of relatively small moldings, the amount dispensed can be checked by weighing the finished part (taking account of the air displaced and any gas loss).

Since other tests involve cutting open finished moldings, they can only be carried out on a limited number of specimens. These tests should be carried out 24 hours after demolding at the earliest.

Samples should be taken from several points on the foam article produced (particularly at points which have proved critical) and checked for cell structure, cell size and density. It is important to take samples from the beginning and end of the flow path taken by the reaction mix. The density in the core (core density) of a foam is lower than the overall density at any given point of the flow path, due to compaction at the skin zone. It is vital to ensure that a certain minimum density is maintained throughout the core. This minimum density is established by the need for dimensional stability: depending on cell

structure, cell size, number of open cells, formulation and type of blowing agent, foams can shrink at low temperatures or expand unduly at high temperatures. Foam samples or whole moldings are therefore checked for dimensional stability for 3 hours at -30 °C and 5 hours at +100 °C.

It is often necessary to test foams for adequate adhesion to facings.

As objective testing is time consuming and involves determining the transverse tensile strength (cf. DIN 53292), a simple process has been devised for fast production control, i.e. trying to pull the facing from the foam by hand. If foam residues remain on the facing after it has been pulled off, the adhesion is good. The force required to pull the facing off is also recorded and compared. It should be noted that this is a subjective, comparative test which does not allow any conclusions to be drawn with regard to the function of the sandwich panel, unless it is possible to prove by careful testing that a certain pattern of foam tearing can be defined as representing adequate adhesion.

The quality controls mentioned are adequate in most cases for detecting faults in time and avoiding subsequent damage. In special applications such as low-temperature insulation, district heating pipes, sandwich panels and spraying, specialized application-oriented testing is advisable.

A rapid and convenient method of checking the dimensional stability of in-situ foam involves taking samples for measuring after 20 minutes with repeat measuring after 1 hour and 24 hours.

For applications with extreme temperatures, foams require testing at appropriate intervals at service temperatures. In the case of sandwich panels, flatness, thermal expansion and buckling strength should be tested. Layers of foam produced by spraying should also be tested for adhesion to the substrate and inter-laminar adhesion.

5.2 Rigid polyurethane foam dust

The fabrication of rigid polyurethane foams often involves sawing, milling and grinding leading to considerable amounts of dust. This dust, like many other dusts, is not only unpleasant for the people involved in the finishing processes but can also irritate the lungs and respiratory tracts. There is also a danger of dust explosions.

To avoid irritation and health risks, the following measures should be taken in works where rigid foam dust is generated:

1. Adequate exhaust ventilation should be provided at each place of work to remove the grinding and sawing dust.
2. People entrusted with work of this type should, if possible, wear half-masks with appropriate filters.
3. The workplace should be cleaned at least once a day - or more often if large amounts of dust are produced - with a suitable industrial vacuum cleaner.

Attention is also drawn to the breathing protection leaflet produced by the German Chemical Industry Trade Association:

Carl-Heymanns-Verlag KG,
Luxemburger Str. 449,
50939 Köln,
Tel.: (+49) 2 21/94 37 30,
Fax: (+49) 2 21/94 37 39 01.

Rigid foam dust, like all combustible dust, can form explosive mixes with air. The accumulation of dust in dangerous amounts should be avoided (1 mm thick layers are considered dangerous). The explosion guidelines produced by the Chemical Industry Trade Association should be consulted.

5.3 Fire precautions

At this point there is no need to go into the fire performance of foams in terms of the official regulations relating to building supervision and insurance law.

All organic materials, including polyurethane foams, are combustible, so appropriate precautions have to be taken for processing and storing in accordance with regulations laid down by the official authorities.

Fresh foams may occasionally self-ignite in unfavorable conditions and when metering mistakes have been made, so freshly made foams should be watched carefully.

If polyurethane foams should catch fire, proven extinguishing agents are: water, water and foam, ABC or BC dry powder (in accordance with DIN 14406).

Carbon dioxide extinguishers of a conventional type do not work well. Breathing apparatus should be used when extinguishing fires in enclosed spaces.

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The methods described in this publication for testing the fire performance of polyurethane, and the results quoted, do not permit direct conclusions to be drawn regarding every possible fire risk there may be under service conditions.

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