Efficient Preparation of Ceramic Feedstock with Compounding and Granulation in the Eirich Intensive Mixer

Introduction

In the preparation of ceramic feedstocks for the production of microcomponents, a difference is made between low-pressure injection moulding, also known as hot moulding, and high-pressure injection moulding. Whereas low-melting paraffin waxes with a melting point of 50 – 70 °C are used in low-pressure injection moulding feedstock, in the preparation of high-pressure injection moulding feedstock a wide range of thermoplastics, like, for example, PE, PP, PPS, POM or even thermoplastic blends are added. The melting points of these thermoplastic materials range from 110 – 170 °C, so, to melt these, a much higher temperature level is necessary than for low-pressure injection moulding feedstock.

Whereas in the preparation of low-pressure injection moulding feedstock, jacketed double-arm mixers, agitators, which are used to stir the powder blends into a melt, or even heated ball mills are used [1], for the production of high-pressure injection moulding feedstocks, according to *Kollenberg* [2] and *Moritz* [3], essentially kneaders and extruders are used. The reason for this is the much higher viscosities of the solids-filled compounds. As the mixing and dispersion efficiency of the kneaders is limited owing to their low-speed tools, the homogeneity of feedstocks prepared in kneaders is frequently only satisfactory [2]. Moreover the throughput rate is relatively low and a relatively high quantity of particles is abraded from the complex-shaped kneading tools, which are consequently difficult to protect against wear [12]. Especially in the case of ceramic products, this leads to unwanted greying or defects in the finished product caused by abraded iron particles.

To improve the homogeneity of the mix in the kneaders, a wide range of mixers is used upstream, simple tumbling, V-mixers and turbulent mixers mainly being used. The achievable homogeneity of the dry materials mix is, however, also severely limited because the low shear forces that can be introduced into the powder mixes with these mixing systems are not sufficient for complete deagglomeration of the usually very fine-grained powder agglomerates. Plasticizing is not possible at all with such mixing systems.

Fig. 1
View into a 150 l EIRICH mixer in the process of preparing high-plasticity ceramic bodies for extrusion

As inhomogeneities in the feedstock cannot be rectified in the downstream processing steps [2] and these lead to defects in the finished product [3], optimum homogeneity, and especially constant homogeneity over multiple batches, is essential for the production of high-quality components. As the fine-grained starting materials are generally prone to strong agglomeration, sufficiently high shear forces and in some cases the addition of dispersants are necessary for the complete liberation of these materials. Sufficiently high shear forces cannot be generated with most mixing systems so that dispersion and surface wetting of the solid surfaces with binder are performed over long preparation times in kneaders and extruders. The upstream mixers are used merely for coarse premixing the raw material components. Subsequent preparation in heated kneaders and shear roller extruders is frequently performed in several sub-steps with precompounding with a sub-quantity of the solid, concentration based on successive powder addition over many hours and, if necessary, several stressing and granulation cycles in a shear roller extruder [4].

On account of its improved homogeneity compared to kneaders, the lower abrasion as well as its easier cleaning, the shear roller extruder is currently regarded as the most suitable equipment for the preparation of feedstocks [2]. However, the throughput rate of theses relatively expensive machines is relatively low. In addition, segregation problems caused by the powder floating on the wave of fluid or the detachment of the "skin" from the roller are known [4]. Sometimes the homogeneities required can only be achieved by multiple recirculation.

To offset the shortcomings of the kneaders and extruders, these are often run with increased binder content. The additional binder is added to achieve a sufficiently low viscosity and therefore sufficient mobility of the compound in the kneader, so as to guarantee the necessary homogenization and wetting of the solids.

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Fig. 2
Receiver for the mix of dry materials and thermoplastic

This conflicts with the desire to minimize binder content in the feedstock in order to minimize shrinkage during drying and sintering.

The feedstock is granulated with blade granulators [12] or by extrusion of the plastic body through a perforated plate with subsequent hot cut-off of the extrusion string.

Preparation in the EIRICH Intensive Mixer

In the following, innovative preparation possibilities beyond the state of the art are described. Core element here is the EIRICH intensive mixer, which with its dedicated design principle, rotating mixing pan and its eccentrically arranged mixing tool is exceptional in respect of efficiency and flexibility among the known mixing systems. Thanks to the separation of material transport with the rotating mixing pan and stressing of the material with the mixing tool, the geometry of the mixing tool is freely designable. Its extremely wide speed range with circumferential speeds from 1 to 30 m/s is also freely selectable and can be easily adjusted in the different process phases. The small tool diameter relative to the diameter of the mixing pan allows it to manage even extreme torques in high-viscosity mixes.

The EIRICH intensive mixer, which has already been used in the ceramics industry for many decades, has the ability to process excellent dry material mixes as well as extremely fine-grained material systems and granulate structures to high-plasticity bodies all in the same machine. Selection of the right operating parameters and operating mode are the only factors crucial to success. This special feature of the EIRICH mixer now enables users to combine the complete feedstock preparation in a one-pot process, as already shown for low-pressure injection moulding feedstock [1] and now also extended to high-pressure injection moulding compounds.

The preparation steps, which in this case can be combined in one single machine, begin with the dry mixing of all powder starting components as well as the thermoplastics or thermoplastic blends (Fig. 2) as well as any necessary liquid or paste-like additives. Mouldings produced during the start-up of the injection moulding machine and/or rejects from the injection moulding process can also be added to this raw material mix. Pre-comminution is not necessary for small-size components. The thermoplastics do not have to be finely comminuted either – plastic granulates or coarsely comminuted binders can be added without any problems.

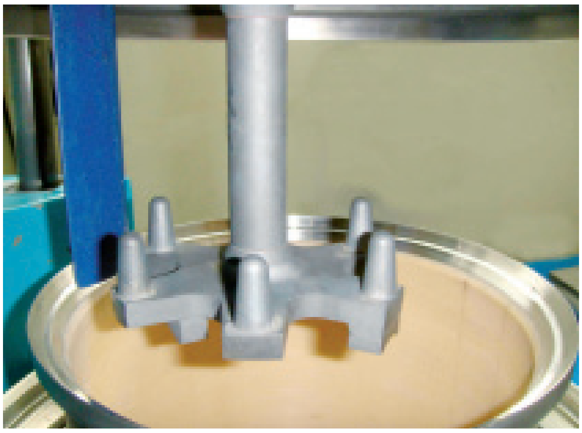
Whereas in other mixing systems, the thermoplastics must be melted separately, in the EIRICH intensive mixer they can be heated simultaneously with the heating of the dry materials. Depending on the thermoplastic used and accordingly the necessary temperature level for the melting process, the heat generated by frictional forces between the tool and material mixed is already sufficient. If the associated preheating times are too long, the preheating process can be accelerated with additional external heating. The frictional heat input into the product depends, for example, on the selected tool geometry and the direction of rotation of the mixing tool. Whereas for optimal dry homogenization with simultaneous effective heating, a star-type rotor with blades protruding radially from a vertical shaft, at a slant to the rotating plane is useful, tools extending downwards and working co-currently with the mixing pan can introduce very high shear forces into the material mixed. This process of dry deagglomeration of the powder starting components can be enhanced with the application of vacuum in an EVACTHERM® mixer. In this case, as a result of the removal of a large part of the disturbing gas phase, the bulk density of the raw material is increased and accordingly the energy input and shearing effect of the tools are increased.

In addition, it has been established that frictional heat depends heavily on the bulk density of the raw materials used. While heating of a ceramic mix has been established in the range of 1 – 2 K/min, for metal powder injection moulding feedstock with bulk densities up to 4 kg/l heating up to 25 K/min solely generated by frictional heat can be observed.

Especially in the case of fine and ultrafine material systems to nanoparticles, sufficiently high shear forces are necessary to overcome the interparticle adhesive forces and achieve complete dispersion. *Daumann* compared different intensive mixers, for instance for the dry dispersion of nanoparticles and showed that, compared to the other mixing systems he investigated, a considerable leap in homogeneity in the EIRICH mixer was possible from circumferential speeds of around 2000 min^{-1} [5]. Further increases in speed in the EIRICH mixer led to a further improvement in dispersion of the nanoparticles and therefore better homogeneity [5].

Owing to their low power input, simple mixing systems frequently used in powder injection moulding, such as tumbling and ribbon mixers, leads to far inferior final homogeneities than those achieved in an EIRICH intensive mixer, as *Rikken* showed on the basis of fine-grained glass mixes in extensive field studies [6]. These maximum achievable final homogeneities cannot be improved with longer homogenization times, as the required local shear forces for the liberation of agglomerates are not available.

In the EIRICH intensive mixer, shear gradients of almost any intensity by adjustment of the mixing tool speed and rotational direction can be fixed. Thanks to the dedicated design principle, the greater danger of contami-



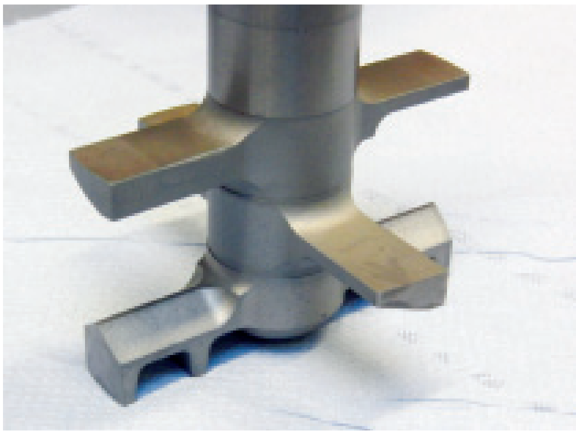


Fig. 3
Mixer with solid carbide studded-type rotor and pan lining made of polyurethane (left) – solid carbide star-type whirler (right)

nation caused by tool abrasion with increasing speed can be countered with the appropriate selection of high-performance materials. Whereas the complex geometries of the tools used in kneaders make it difficult to use, for example, ceramics and/or carbides, the simply shaped tools in the EIRICH intensive mixer can be hard-faced with ceramic or carbide materials. To guarantee maximum service lifetime and minimal wear, the entire mixing tool can be made of solid carbides (Fig. 3).
To minimize the possibly still problematic abrasion from pan walls and/or wall scrapers, these can, especially in the case of low-pressure injection moulding compounds with a maximum product temperature of 80 – 100 °C, be lined with suitable temperature-resistant plastics. For the higher-melting thermoplastics with temperatures over 170°C, it is no longer possible to reduce material abrasion with plastics; here, for instance SiC ceramics, owing to their good thermal conductivity, or other ceramic materials can be used. However, the abrasion from mixing pans and wall scrapers so far observed has been so low that they do not lead to feared greying of the finished product and/or defects in the sintered product.
As *Gerl et al*. has already shown for low-pressure injection moulding compounds, with the use of the Eirich mixer, preparation time could be reduced, compared to traditional processes, from several hours to just a few minutes [1]. On account of the very short preparation times achieved, for low-pressure injection moulding compounds, generally no additional heating equipment is necessary to further accelerate the preparation process. The processing/melting of the thermoplastics in high-pressure injection moulding compounds could generally be effected solely with frictional heat, but considerable time reductions could be achieved with an external supply of heat. Supplementing the mixer with suitable heating equipment therefore appears a sensible option. For reasons of possible contamination by the high-speed mixing tool with the much longer preparation times for high-pressure injection moulding compounds alone, the use of external heating is recommended to reduce the times required.

Heating Methods for Rotating Mixing Pans

To achieve efficient heating of the powder mix with the high-melting thermoplastics, the supply of additional heat through the mixing pan wall or the base, as described above, is expedient. With the rotation of the mixing pan in the EIRICH mixer, heating by means of thermal oils in a double jacket can be essentially eliminated as an option so that two different non-contact heating methods are applied to heat the rotating mixing pan. Besides radiation heating, which has been used for decades and undergone continuous optimization, induction heating, which has been used in industry for several years, boasts key advantages for everyday production operations.

Radiation Heating:

Especially for laboratory operation or occasional production operations, the rotating mixing pan can be very efficiently heated with low-cost quartz lamps. Besides very high efficiency, these feature the advantage of very short switching times and low inertness compared to the flexible tubular radiators that have been used for many decades. The rotating pan is radiated linearly by a detachable quartz lamp heater and is therefore uniformly heated over its entire circumference (Fig. 4).
The radiated heat is then given off into the material mixed in the mixing pan via thermal conduction within the mixing pan walls. The coefficients of thermal conductivity achievable with this constellation can be much higher than that known for comparable mixing systems. This leads to noticeably shorter preheating times, which are generally only limited by continued energy supply from outside. Especially for scale-up, the arrangement of several parallel-connected quartz lamp heaters can transfer sufficiently large quantities of energy into production machines. With the short switching times and accordingly the fast response characteristics of the quartz lamp heaters, a very good temperature control is possible based on the monitoring of the outside pan temperature with pyrometers, and the temperature of the material being mixed measured directly with temperature sensors.

Induction Heating:

The inductive heating of rotating mixing pans introduced into industrial practice several years ago boasts clear advantages especially in the harsh conditions of everyday production. As with induction heating the heat is generated directly in the mixing pan wall and does not have to be supplied from the outside with flowing or radiating heating media, extremely high heating rates can be realized with excellent control on the available pan area.
Through a very small heat input area, the inductor heats the mixing pan uniformly through its thermal insulation over the circumference of the pan. Over the entire jacket surface the inductor gives off its heat into the material being mixed inside. The heat transfer coefficients to the material

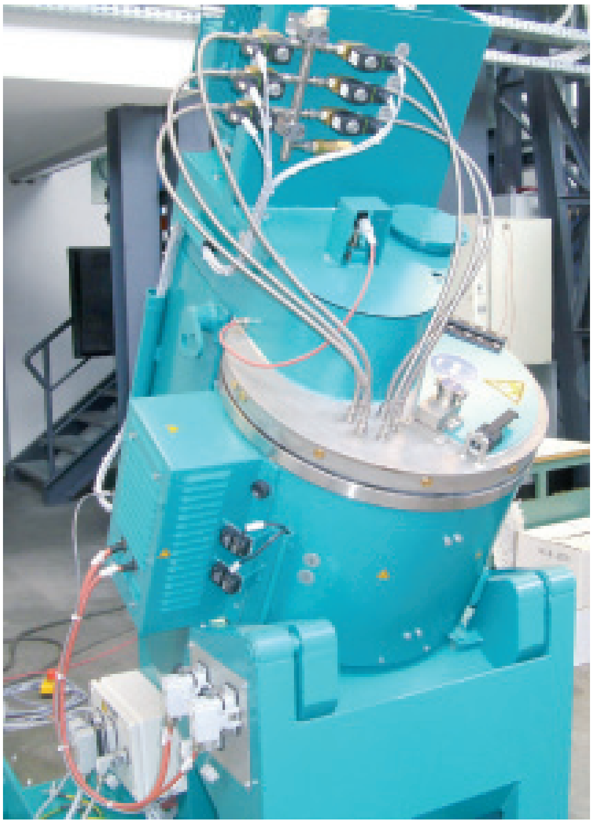


Fig. 4
R05T mixer for plasticizing and granulating paraffin-containing mixes with quartz lamp heaters and CO_2 liquid gas cooling

mix are comparable with that of radiation heating while the energy supplied in the extremely small and compact unit is much higher than possible with quartz lamp heaters. In addition, thanks to modulation and extremely short response behaviour, the temperature can be easily and effectively adjusted based on control of the power supply. Both heating systems enable wall temperatures in the range from 200 to 300 °C, so that very high preheating rates can be realized for high-pressure injection moulding compounds. On account of the much higher heating rate per unit of area possible with induction heating, this can be used to achieve even higher preheating rates than those realized with quartz lamp heaters. The preheating rates achievable with this system are far beyond than those possible with liquid-heated systems, as here the maximum possible heat transfer to the wall is limited by the flow rate and the pressure loss in the double jacket.

Plasticizing and Kneading in the EIRICH Intensive Mixer

As mentioned above, preparation in the EIRICH intensive mixer starts with the feeding of all dry materials and all thermoplastics as well as any liquid and/or paste additives. Subsequent dry mixing and heating of the mix is done at mid-range tool speeds so as to avoid excessive dust generation. In this respect, the simultaneous application of vacuum in the EVACTHERM mixer showed a substantial reduction in raised dust. During the mixing process, the compound is heated with the frictional heat transferred by the tool and through the additional radiation or induction heating. A typical preparation cycle is shown in Fig. 6.

Depending on strength and size, the thermoplastics or moulding waste added are effectively comminuted by the mixing tool and accordingly they can be added in relatively coarse lumps. With many thermoplastic binders or additives, a sticky effect develops from a certain temperature, which leads to spontaneous and complete binding of any dust into the compound. This softening point is often reflected in slight increases in the power consumption of the mixing tool and is a good indicator for the subsequent control of the production process. For example, with a subsequent increase in speed without dust generation, it is possible to accelerate preheating. From temperature curves, it is possible to derive information on the material behaviour, as during melting of the thermoplastics the melting heat is absorbed, generally leading to a flattening of the temperature increase or inflection points. This is accompanied by a steep increase in motor power caused by the plasticizing of the material. Owing to increasing adhesion forces caused by the melting of the thermoplastics, the proportion of frictional heat in the heating process increases until the thermoplastics contained in the mix are completely melted and the motor power falls substantially again as a result of the decreasing viscosity with rising temperature. In this case phase of varying viscoplasticity is formed depending on the binder content and type, which especially at the melting point effects maximum power input into the mix. The correct selection of drive concept and adequate motor rating are crucial for trouble-free kneading in the mixer.

The processing of any binder concentration is, as many tests have already shown, no problem in the EIRICH intensive mixer. The shear stress can also be adjusted depending on the selection of the tool speed so that according to *Wu*, optimal disintegration is achieved [8] and no degradation of binder components caused by excess stress occurs, like sometimes observed in kneaders and shear roller extruders [3].

As the thermoplastic is already present in the mix over the entire preparation time, the well-dispersed particle surfaces are completely coated with the thermoplastic. The tests also showed that, following preparation in the EIRICH intensive mixer, the viscosity was at least just as low as or even lower than the viscosity obtained after preparation in a

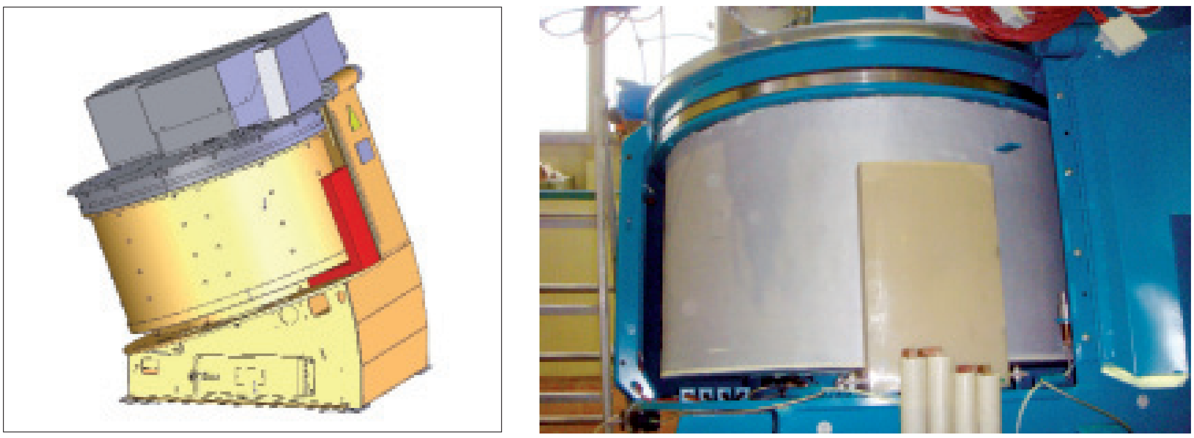
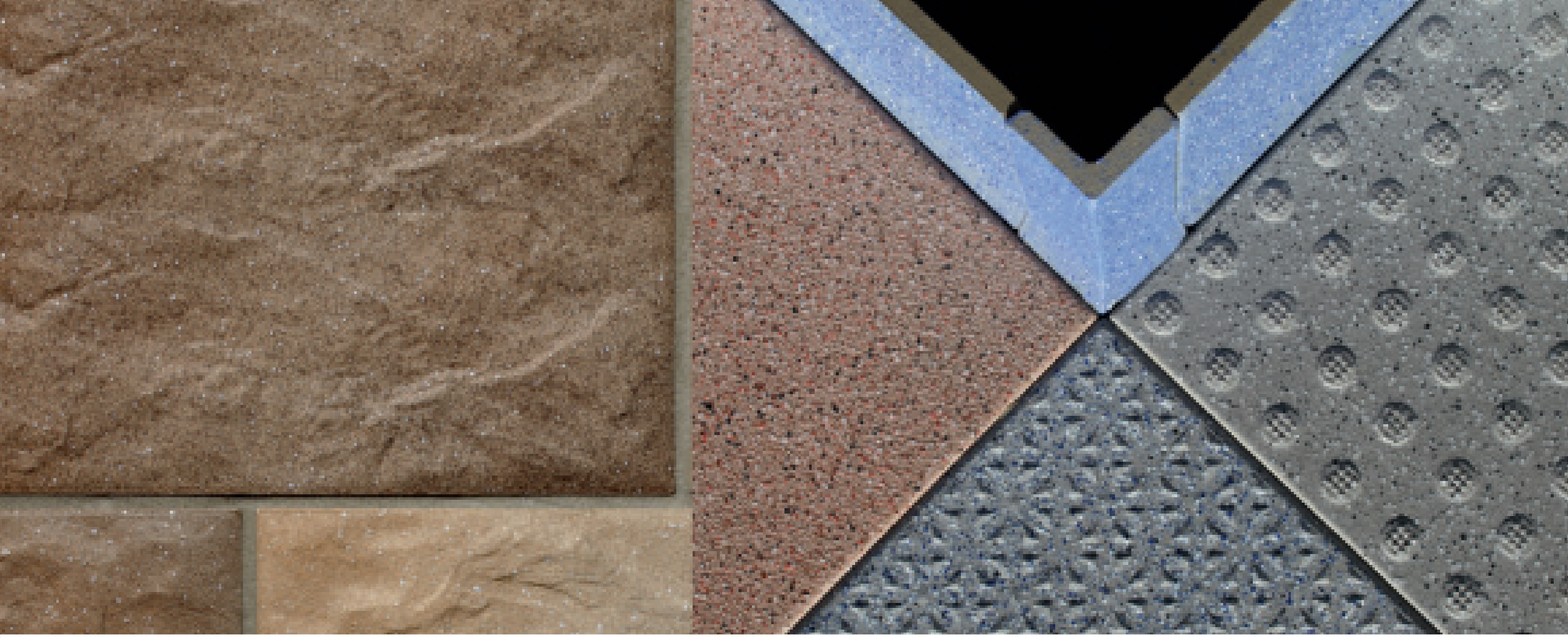


Fig. 5 400-l R12W mixer with L-shaped 160 kW inductor and directly mounted pan insulation for mixture temperatures to 260°C



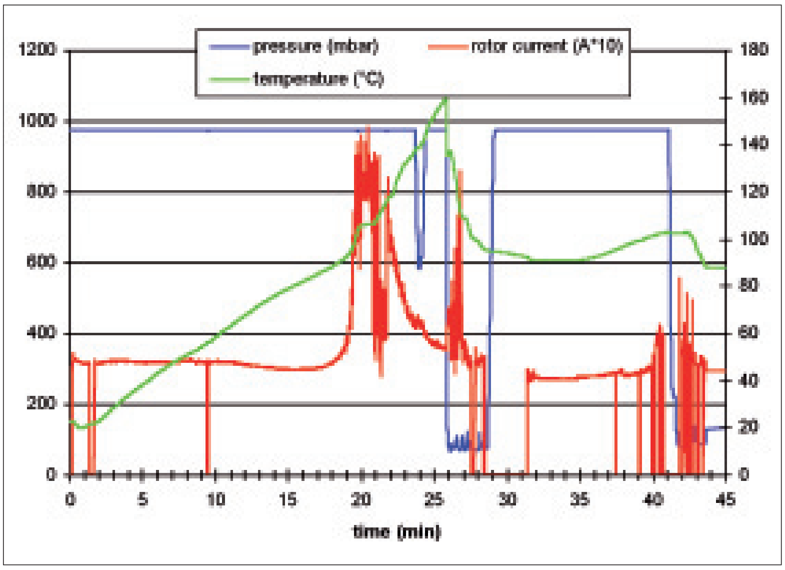


Fig. 6
Preparation of high-pressure injection moulding feedstock – time curve of the mixer pressure in the EVACTHERM® mixer, temperature of the mix and motor power

kneader, which already *Suri* and *de Souza* attributed to the better disintegration of the agglomerates and the resulting improved packing efficiency in the powder [9, 10]. In the tests conducted, the effect of the application of vacuum proved particularly positive with regard to disintegration of powder agglomerates.
The compound produced after plasticizing must be only subsequently remixed for a short time as generally complete deagglomeration and coating of the solids surfaces by the thermoplastics have already taken place in the starting and melting phase of the mixing process. If the homogeneity of the mix in this state is not acceptable, the temperature in this phase can be easily kept constant with appropriate cooling methods and more intensive shearing of the plastic body effected.

Cooling Methods for Rotating Mixing Pans

As shown above, with progressing coherence of the compound, the frictional heat becomes the dominating effect in preheating; this can be a problem particularly in the plastic phase. Here continued heating leads to the permissible temperature limits of the thermoplastic binder being exceeded or to a decrease in viscosity, which conflicts with improved dispersion [8]. To control the temperature in these phases, effective cooling is necessary. All wall-cooled systems have the disadvantage that the heat flow through the existing heat exchange area is limited owing to the relatively low temperature differences, which has adverse consequences particularly on scale-up. In addition, material build-up at the wall further reduces heat transfer. While preparation in small-volume kneaders and mixing pans can be performed wall cooling without any problems, on subsequent scale-up to production-size machines, it is hardly possible to control the temperature curve in this way. Especially if the mix is to be heated from the outside through the walls of the mixing pan and then the temperature is to be controlled by cooling, the same machine would have to be switched from heating to cooling, which would be a very long drawn-out process owing to the inertness of the mass of the machine. Consequently, series-connected heating and cooling mixers are often used.
To enable scalability of the EIRICH intensive mixer to any size, the following long-established methods of evaporative cooling have been applied in order to avoid the problem described above. Especially for the high-melting thermoplastics and mix temperatures in the range from 150 – 180 °C, cooling can be effected in the simplest case with the addition of liquid onto the product surface under atmospheric conditions. The liquid evaporates spontaneously on contact with the solid surface and, on account of its latent heat, very efficiently extracts the heat from this surface. As extremely good material and heat transfer takes place in the EIRICH intensive mixer on account of the turbulent material movement, with the controlled spraying of, for example, water onto the product surface, a very good temperature control is possibly. Fears that the water could adversely affect the product properties owing to the hygroscopicity of, for example, alumina are unfounded as *Novak*'s investigations have proven. Water tended to have a positive influence on debinding and led to an avoidance of defects in the finished product [11].
This was also proven in tests on ceramic low-pressure injection moulding feedstocks. The use of a liquid cooling medium for evaporative cooling had no adverse effect on the technical properties at all [1]. In addition, the amount of liquid added is calculated so that complete evaporation from the body or particle surface coated with thermoplastic is ensured.
The evaporative cooling process described above has been used for years in industrial plants with up to several MW thermal power. The liquid mass flow can be controlled as a function of the mix temperature so that the addition of liquid only offsets, for example, the frictional heat introduced by the tool. With this method, preparation in the plastic phase can easily be performed at constant temperature and viscosity over any length of time in differently sized mixers from 5 to 500 l.
As an alternative, the use of liquid gases, such as liquid CO_2 or nitrogen, as a coolant is also possible. The liquid gases are sprayed into the circulated material so that they only come into direct contact with the product and, as far as possible, do not actually touch the machine parts. Thanks to the rotating mixing pan, this can be easily done from the top of the mixer (Fig. 4). The permanent material circulation ensures uniform mixing-in of the liquid gases or the dry ice snow. With this selective pure product cooling, minimal liquid gas consumption can be realized, and above all fast switching from heating to cooling is possible without complete lowering of temperature of the mixing pan itself.
The amount of liquid gas added can, like the amount of cooling liquid, be controlled based on the temperature of the material in the mix if only the frictional heat introduced by the mixing tools must be offset. Especially effective here is the use of liquid CO_2, as sublimation of the liquid CO_2 only takes place in the nozzle inside the mixing pan, enabling very good dosing. A more energy-efficient process is possible with the use of liquid nitrogen, which, however, in the case of intermittent dosing can lead to problems in dosing accuracy already in the supply lines if these are not suffi-

ciently thermally insulated. The liquid gases used for cooling gasify on heating, leaving no residue.

Granulation of the Feedstock

For the last stage in the production of feedstock granulates, various process options are now available. The following are possible:

a) Extrusion with hot face pelletization
b) Evaporative cooling with comminution
c) Evaporative cooling with comminution and subsequent granulation
d) Comminution with blade granulators

To obtain granulates with proven geometry, flow behaviour and size distribution, the plastic body produced in the mixer can be transferred into a downstream, heated extruder with simple perforated plate geometry. In the extruder itself, no further homogenization and heating take place. Only strings of feedstock are drawn, which on exiting through the perforated plate is granulated by means of hot face pelletization.

As an alternative to this, so as to avoid additional equipment costs, complete production of granulates is possible in the EIRICH intensive mixer. For this purpose, extremely fast cooling of the hot compound to below the setting point of the thermoplastics is necessary, while at the same time the high-speed mixing tool comminutes the plastic compound into a granulate-like structure. To effect this fast cooling, cooling with wall-cooled systems can be rejected as an option as these are generally only able to remove the heat introduced into the mix by friction through the wall of the mixing pan. To realize fast cooling, as described in the section on temperature control above, the plastic compound is cooled rapidly with the help of evaporative cooling based on the addition of a liquid or liquid gas. The amount of coolant added direct to the mix can be adjusted to determine the cooling rate and the temperature reduction. The crucial criterion for the selection of the amount of liquid added is that this must be completely evaporated after the cooling process. Generally it is sufficient to choose the amount of coolant so as to remain just under the setting point of the thermoplastics. With further cooling, the granulate becomes increasingly solid and brittle so that the tendency towards further comminution with the high-speed mixing tool and consequently the formation of a powder structure increases. If rapid cooling is performed with simultaneous comminution of the mix by the mixing tool to below the setting point of the thermoplastics, granulates can be obtained in the mixing pan in just seconds (Fig. 7). With the selection of the tool speed and cooling rate, it is possible to influence the granule size distribution.

As tests with a wide range of thermoplastics have shown, the type of thermoplastic and the solids content decide the shape and size of the granules obtained by comminution in the granulation process. If the granule size distribution obtained in this method is too wide, the sphericity of the granules too low or the powder content too high so that flowability in the downstream injection moulding machines is still unacceptable, it is useful to optimize the granulate by subsequent heating to just below the melting point.

Here one possible procedure is first, with high tool speeds and correspondingly low temperatures, to produce more or less a powder product by disintegration of the granules produced after the first cooling phase. These are then regranulated by heating to the melting point of the thermoplastic. As a result, small granulates with a relatively narrow size distribution are produced. Alternatively, coarse granulate with an excessive fines content can be directly reheated so that essentially the fines are regranulated and the granule size distribution shifts to a coarser product (Fig. 8).

Fig. 7 Granulates produced by comminution of the plastic compound with simultaneous cooling

Here it is important that, especially a few degrees before reaching the actual melting point, the temperature input is controlled and preferably the heat only introduced by the mixing tool. In this way, depending on the thermoplastics used, granules with high sphericity can be produced, their size distribution being adjustable as a function of the mixing tool speed. Granulation is effected solely with the softening of the thermoplastic-coated surface of the particles, which then bond with adjacent particles as a result of the adhesive forces and build up to form granules. Here the advantage of the inclined rotating mixing pan comes into play,

Fig. 8 Plasticized, comminuted and subsequently granulated feedstock based on different binder systems (a and b)

with formation and shaping or profiling of the granules being optimized thanks to their rolling motion in the inclined pan. The product granulated in this way can be emptied directly and cooled externally on a cooling surface; alternatively it can be cooled in the mixer with very gentle action of the mixing tool so as to avoid comminution of the formed granules. Besides using water as a coolant, solvents can also be used. Here, however, it is important to ensure that these do not react with the thermoplastic or other constituents. Generally the use of solvents, however, is not as effective as that of water as the evaporation enthalpy is much lower. In addition, special measures must be taken to protect the machine against explosion and the necessary peripheral equipment must be incorporated into the system. Important for all cooling methods involving liquids is that the amount of liquid added completely gasifies and no residue is left in the compound.

Besides the evaporative cooling processes with liquids or liquid gases, the plastic or flowing compound can also be solidified in an external cooler following preparation in the mixer and the plate-like structures produced can then be comminuted. Up to now, this comminution is frequently performed in blade granulators [12]. Alternatively, to avoid investment in additional machinery, coarsely comminuted particles can be finely comminuted in the EIRICH intensive mixer with appropriate tool shearing similar to in blade granulators. The not uniformly shaped material formed, with the high content of fines < 150 µm [12], can be transformed into a granulate structure with spherical granules in a narrow distribution and with a low fines content by subsequent heating in the mixer to close to the melting point. The air inclusions feared with conventional blade granulation, which collect in the cavity of the injection moulding machine and lead to gas bubbles there [12], can be reduced substantially with the use of an EIRICH EVACTHERM® mixer based on comminution in a vacuum.

EVACTHERM® Vacuum Preparation

For particularly expedient preparation, one option is the application of vacuum during the entire process. While, as described above, the application of a low mixer internal pressure has a positive influence on the dispersion of the dry material particles by increasing their apparent density and therefore the shear energy input, especially on transition into the plastic phase and in the plastic phase itself, complete degassing is possible in vacuum preparation. Vacuum preparation results in a much lower content of bubbles in the solidified product.

While the air in the plastic compound works like an non-plastic agent, with the application of vacuum, the viscosity of the compound is apparently increased. On account of the absence of air bubbles, the particles are much more densely packed so that the mixer responds with much higher power consumption. This, however, generates a much higher shear rate in the mix, which in turn can be used positively for the final dispersion of all components.

In the cooling phase, the application of vacuum can also lower the temperature of the mix. Here the vapour pressure curve of the liquid coolant is used, which for water, for example, can lead to product end-temperatures of around 45 °C at 80 mbar pressure. Insofar final cooling to the final processing or storage temperature could be done directly in the mixer. With this aim, the mixer is fitted with a vacuum-proof casing, while the rotating mixing pan corresponds to that of a mixer operating under atmospheric pressure. The necessary peripheral equipment generally consists of a vacuum pump for control of the applied pressure or for fast pressure reduction during the vacuum cooling phase, when otherwise mixing is performed at atmosphere pressure. In addition, the evaporated liquid can be almost completely recovered with an intermediary condenser.

Premixes and Microgranulates

For many customers, the idea of replacing a multistage process with a one-pot process with much shorter preparation times and much less handling is too drastic for their everyday production operation. Here possibilities like those described above are available to optimize installed preparation plants.

Before the compound heated with frictional heat and externally supplied thermal energy reaches a completely plastic state, a few degrees below the melting point of the thermoplastic, a granulate-like, dust-free structure is formed, which contains excellent homogeneity thanks to efficient mixing of the dry constituents and boasts excellent distribution of the thermoplastic on the surface of all particles. The caviar-like structure obtained in this way (Fig. 10) is very suitable for feeding to downstream kneaders or shear roller extruders, to combat any segregation processes occurring in these or deficiencies in the mixing of the dry constituents.

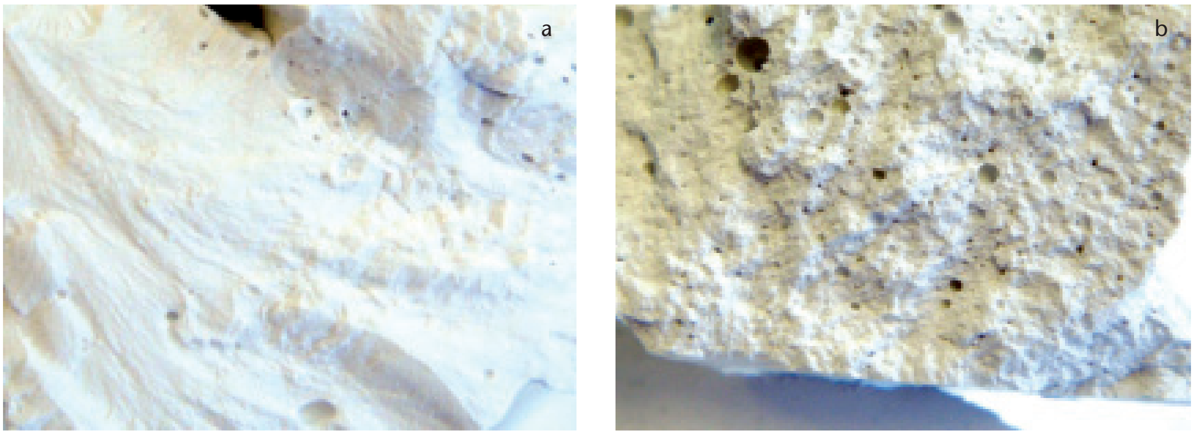


Fig. 9 Comparison of injection moulding compounds with (a) and without (b) preparation in vacuum

Especially as a result of the pre-heating close to melting point already realized in the mixer, the compound can be transferred completely uncooled to the downstream kneading step so that the throughput rate of the installed kneaders or extruders can be increased thanks to the much shorter heating and kneading times. This procedure would also have a positive effect on contamination of the prepared compounds with particles abraded from the kneading paddles; in addition the homogeneity of the final products is clearly improved thanks to the homogeneous dry material mixing and effective dispersion.

Fig. 10 Fine-grained granulate structure obtained by melting the raw materials mix without plastic phase

Summary

As the observations above have shown, the use of an EIRICH intensive mixer enables technically much simplified and efficient preparation of ceramic high- and low-pressure injection moulding feedstock. This novel preparation technology has already made inroads into industrial practice. Besides the much improved homogeneity and reduction of problems encountered with established preparation machines, the combination of several process steps (heating, plasticizing and granulation) in just one single machine gives considerable potential for cost saving.

With the selection of appropriate wear materials, contamination by metal abrasion can be minimized; in addition preparation times lasting many hours can be reduced to a few minutes. Compared to many kneading and extrusion systems, the investment costs are reduced considerably, so that besides lower cost production, greater flexibility in the production of different formulations is possible. Thanks to the feed consisting of all dry materials as well as thermoplastics and additives, the complete formulation is already contained in the total mixture from the very first second. The simplified preparation procedure reduces potential sources of error, for example as a result of the later addition of binders or dry materials, which is frequently done manually, tying up labour resources at the machine. With intelligent monitoring of the power consumption of the mixing tool and the temperature, it is possible to automate the entire preparation cycle.

All raw materials are comminuted and dispersed with appropriate, adjustable shear stress. As a result of heating with the frictional heat and additional heating of the mixing pan walls, which can be effected with radiation heating or – particularly expedient – inductive heating, in a short preparation time, a highly plastic compound can be obtained by melting the thermoplastics. Once a plastic compound is obtained, dispersion and complete coating of the particle surfaces are already largely completed so that only short rekneading is necessary in the plastic phase. If longer kneading phases are necessary, these can be completed at constant, controllable product temperature and therefore viscosity with the help of suitable evaporative cooling technologies. Here the entire mix is kept at a constant temperature level, without any temperature gradients between the mix and the pan walls. With evaporative cooling with liquids or liquid gases under atmospheric pressure or alternatively with the application of vacuum, the entire plastic compound can be cooled very rapidly. When the setting point is reached, the compound is comminuted by the mixing tool into a granulate structure. On subsequent further cooling and/or heating of this granulate, its granule size distribution and sphericity can be influenced. Preparation in a vacuum has the added advantage that air inclusions in the forming agglomerates are completely removed, which results in a lower viscosity of the compound. This special principle of preparation also allows – if required – a reduction in binder content, ensuring lower ma-terial shrinkage and porosity in the finished product. On account of the exceptional variability of the EIRICH mixing principle, further optimization of the material and processing properties can be achieved with the selection of appropriate parameters.

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