Process for the separation of tetramethylsuccinatedinitrile from the exhaust air of plastics processing machines

Introduction

Processing plastics in extrusion plants is connected with the occurrence of considerable amounts of deposits in air evacuation ducts, which can lead to severe malfunctions of the extrusion process and, as a result of this, increased servicing expenditure and downtimes. It was the target of a cooperation project between HTWK Leipzig and the companies Barlo Plastics Nischwitz GmbH and UGT 2000 GmbH to carefully examine these deposits and to develop a suitable procedure for separating this substance immediately in front of the exhaust air cleaning system proper.

The main component of deposits in the exhaust air cleaning system was determined to be the toxic substance tetramethylsuccinatedinitrile (MSN), the generation of which is associated with the use of initiator 2,2'-azo-bis(isobutyronitrile) (AIBN) in the polymerization process. AIBN is utilized in numerous plastics, such as polymethylmethacrylate, polyvinylchloride, or in styrene polymerides, for initiating polymerization, during which, besides radicals, TMSN is created in a side reaction. This byproduct is deposited into the plastic matrix and is partially released during further processing of plastics via exhaust air.

One property of this substance is its sublimation under normal pressure at 172°C (341.6°F). For that reason, TMSN crystallizes predominantly in the cooler regions of air evacuation ducts where, together with other condensation products, it forms thick pasty deposits.

Material properties of tetramethylsuccinatedinitrile

According to TRGS 900, the admissible volume-related limit value for TMSN in air is 0.5 mL/m³ and the mass-related limit value is 2.8 mg/m³. For comparison, the volume-related industrial threshold limit value for hydrocyanic acid in air being 10 mL/m³ is 20 times as high.

These figures illustrate the toxicity and the potential hazards of tetramethylsuccinatedinitrile. This substance causes especially severe intoxication symptoms, which are comparable to the effect of a centrally effective convulsant agent.

Hazards arise during the processing of plastics particularly through the release of TMSN in exhaust air and on direct contact with TMSN crystals when air evacuation ducts are being cleaned. In can be assumed, however, that hazards due to the direct contact with plastics, e.g. for foodstuffs, may be neglected based on low TMSN migration tendency.

Analytics

By means of the analytic processes elemental analysis, IR spectroscopy, and thermal analysis, it has been possible to definitely identify the crystals deposited during the manufacture of plastic plates made of polymethylmethacrylate (PMMA) in the exhaust gas cleaning system of an extrusion plant as TMSN.

The use of appropriate analytical processes for detecting TMSN in exhaust air and in solvents was a necessary prerequisite for this examination. A gas chromatographic analytical process using a flame ionization detector (GC-FID) has been developed for this purpose.

By means of this measuring technique, TMSN concentrations ranging between 225 mg/kg and 360 mg/kg have been detected in granulates of a rubber-modified polymethylmethacrylate.

Laboratory Tests

A laboratory plant was set up for developing a suitable procedure for separating TMSN from exhaust air, which enabled air to be charged with TMSN in a defined way and thus a precise balance of TMSN deposition. During an initial series of experiments, absorption tests using polyglycol-based detergents were carried out.

The drawbacks of absorbing TMSN by means of polyglycols are in particular procurement costs and necessary detergent regeneration in a separate process stage. That is why TMSN sublimation was studied in a second series of experiments on the basis of direct cooling by means of water as the coolant in the exhaust air flow, which enabled separation rates of >80% to be achieved.

Field Tests

Field tests were carried out on an extrusion plant of Barlo Nischwitz GmbH, equipped with a vacuum degasifier. The granulate throughput of this plant was about 500 kg per hour during the test period. Pilot plants for exhaust air pretreatment were arranged directly behind the vacuum pump.

Absorption experiments were done using a bubble washer and succeeded in proving that TMSN can be safely separated from exhaust air by means of this process variant even under real operating conditions.

Because of the drawbacks of the absorption process already mentioned, subsequent examinations focused on TMSN deposition by sublimation. To this end, UGT 2000 GmbH designed and constructed a cyclone on the basis of previous calculations.

Based on analyses carried out and on corresponding weight assessments, it was proved that TMSN separation rates of more than 99% can be safely achieved using this process and the plant systems developed. Besides TMSN, about 20% of monomers contained in exhaust air are also separated by means of the sublimation procedure. This side effect relieves the downstream parts of the exhaust air treatment plant proper, which particularly consists of a biofilter with Barlo Plastics Nischwitz GmbH. After separation, TMSN is present in a MMA-water mixture, which can be disposed of via existing disposal routes.

Different plant variants are available for separating monomer/initiator residues; they can be selected for the individual plant after field conditions have been cleared up.