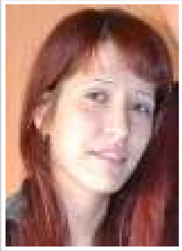
PROFESSIONAL PAPER / STRUČNI RAD

ANALYSIS OF THE CAUSES OF THE OCCURRENCE OF AN IRREGULAR PROCESS OF OBTAINING POLYAMIDE 6

ANALIZA UZROKA NEPRAVILNOG PROCESA DOBIJANJA POLIAMIDA 6

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ABSTRACT

An analysis of the possible failures of the process of obtaining polyamide 6 by polymerization of caprolactam in VK – pipes, by using the FTA method, is performed in this paper. The main factors contributing to the poor progress of process was identified by using deductive analysis and by forming a fault tree for the adopted top event. Considering the possibility of their control, can be reduced the expected losses, and achieved in that way better production of sintered polyamide 6th. The procedure of forming the minimal cut sets for the concrete example is explained. By analysis of the common cause ranking of secondary causes of basic fault tree events, according to their influence on the top event occurrences, was performed, and the possibility of eliminating the causes of failure was considered. In conclusion, the paper presents possible applications of the achieved results.

Key words: *FTA, polyamide, polymerization, VK-pipes, qualitative analysis.*

1. INTRODUCTION

Reliability analysis of technical systems is based on failure analysis of their elements. Alarge number of methods for the failure analysis of the technical system's elements have been developed, among which the most commonly used are Fault Tree Analysis - FTA and Failure Modes and Effects Analysis - FMEA [1,2,3]. These methods can be applied both in the development phase, but also during exploitation of technical systems.

FTA is a deductive method, where at first, the so-called top event, which in the technical systems represents a failure, and then the possible causes of this failure inside the system are analysed. The basis of the fault tree represents a transformation of physical systems to structural logic diagrams.

Fault tree analysis method was invented and developed in the United States in the early sixties of the 20th century [2,4]. From its beginnings until today, the FTA method is widely used for failure analysis, safety research and for failure's diagnostic of a large number of complex technical systems. This method is especially convenient for the reliability and safety analysis of the systems whose failures might cause catastrophic consequences for mankind and environment.

Causal definition of the system's state that leads to failure can be used for assessing the maintainability and for development of the project's maintenance of technical systems. The fault tree can be used, in the exploitation phase, as a diagnostic tool for determination of the most likely cause of the resulting fault event. The application of FTA method is best performed as a product development team activity. Even though an individual may attempt the FTA, the trees that are developed by a team are generally more fully defined and complete. The reason for this is that a much broader sphere of information is presented and considered by a team.

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Due to the complexity of the approach and scope of the outcomes, FTA methods can be classified in the group of indispensable methods in providing quality of technical systems.
Materials which are obtained based on high molecular compounds (polymers) are called plastic masses [5,6]. Thanks to its good qualities, plastic masses are now used in all areas of human activity. Manufacture of the plastic masses increases significantly every year. According to some data, the production of plastic masses exceeds the production of all metals together. Therefore, ratio-nalization in any phase of the production process of obtaining plastics or any product made from plastics, gives the opportunities for huge savings.
The results of FTA method's application in the process of polymerization polyamide VK – pipes are presented in this paper. The aim of this research was to determine the basic factors that contribute to poor progress of the polymerization process.

2. A POLYMERIZATIONOF CAPROLACTAM IN VK – PIPES

Polyamide 6 is obtained by polycondensation of caprolactam, i.e. polymerization of the cyclic amide. Cyclic amides are also called lactams [7].
Polymerization is the creation of macromolecules, by merging of monomers without separation of by-products and the realignment of the atoms in the molecule. Polymerization process must not be interrupted, because it cannot continue in the same way. A special case of the synthesis is the polymerization of various monomers, which is called copolymerization. Thereby, macromolecules consisting of basic ingredients, which are generally repeated regularly in the polymer, are formed. Properties of the resulting polymer are significantly different from the properties of the components (monomers), or the properties of their mixtures.
The polycondensation is the process of forming the long molecules from the same or different monomers with the water residual (condensate). Polycondensation occurs in: phenol, carbamide (urea), aniline, aldehyde [8].
Modified procedure of the polymerization process of caprolactam in a VK - tube with appropriate equipment is schematically shown in Figure 1 [9]. A process of polymerization with a VK - the tube is carried out as follows. Pre-polymerization starts with continuous adding of lactam in the first boiler, which is indicated by *b* in Figure 1. The pressure in the steam boiler is from 2 to 7 bar in the presence of water in the quantity from 0.5 to 4%. In this way, it is achieved 80-85% of the total amount of the product. The temperature in the steam boiler is from 240 to 280°C. Further pre-polymerization continues in the second boiler in a stream of nitrogen, by dropping pressure to the nominal value.

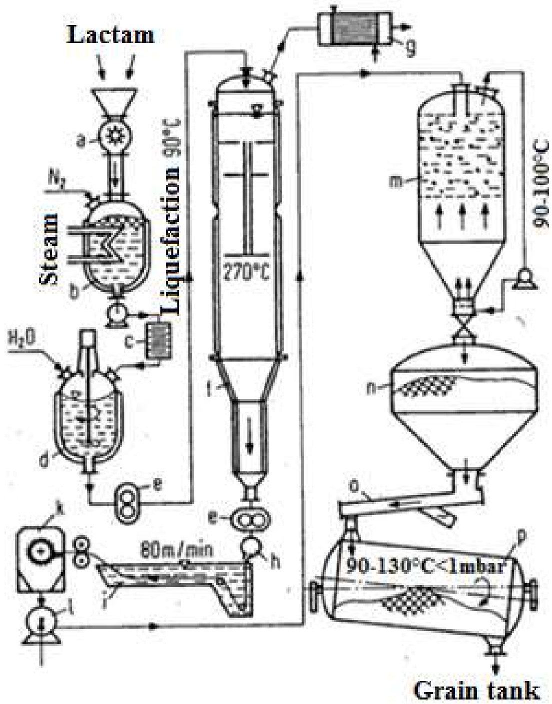


Figure 1. Polymerization of caprolactam in VK – pipes with granulation: a- roller with spikes, b- boiler for melting, c- filter for molten lactam, d- access to the boiler, e- gear pump, f –inner VK - tube, g- capacitor, h- spin bar with nozzle, i- spin bathtub, k- granulator, l- rotary pump, m- extractor, n–granule bunker, o-vibrating tube, p- dryer

The obtainedpre-polymer is by using a gear pump drains into the VK-tube, which is the beginning of the polymerization process. By using a system with a VK tube, pre-polymer is degassed by stirring. With the use of apertures, the reaction mixture is rapidly and evenly heated and degassed. The melt is polymerized while moving down (control of the product's flow through the installation). Depending on the polymerization temperature, the temperature is raised again (temperature control through the heat conductors). A shorter duration of the process is achieved by using a static mixer in the VK - tubes. The usage of high pressure up to 0.9 bar enable increasing of the polymerization temperature and accelerate the polymerization process. The polymerization is carried out in the presence of increased amounts of water under pressure. If the water content is from 2 to 8%, the temperature should not be higher than 280°C (in this case, the temperature is 270°C) and the pressure of 40 bar. In this way, a reducing of the duration of the polymerization process is achieved.

The polymerized product, at the bottom of the VK-pipe, is ejected by the gear pump, which is a beginning of the post-polymerization process. The obtained polyamide 6 is cooled by the water in the spin tub (*i* in Figure 1), and then granulated. The granules are delivered to the extractor by using the rotary pump.

The low-molecular components of poly-caprolactam must be removed. The removal of low-molecular components is conducted usually by extraction granules with water, at the temperatures from 90 to 100°C. Extraction is carried out non-continuously with replacing water 2 to 3 times. The granules are placed into the bunker and put in the dryer, by using the vibrating tubes. The granules are dried at temperatures from 90 to 130°C in a vacuum or with a stream of nitrogen. In this process, continuous heaters are used [9].

3. THE FORMATION OF THE OBSERVED OBJECT'S FAULT TREE

The fault tree analysis procedure includes a number of steps [10,11], and it is fully applicable for the analysis of potential failures of complex technological systems.

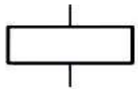
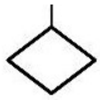
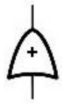
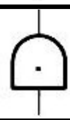
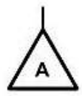
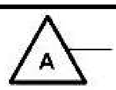
The top event of the fault tree, depending on the analysed system, can be an universal (in the form of a system's failure), or the specific (if it includes only some system's failures or its components' failures).

The formation of the fault tree is done by using the symbols for events, logic gates and transmission [1,2,3,12]. Power of a fault tree symbolism lies in the fact that the symbols for events, coupled by logic gates, can easily be translated into algebraic expressions. The most commonly used symbols for the formation of the fault tree are given in Table 1.

A number of different symbols are using for events that show whether it is a complex or basic initiating events. The rectangle is used for complex events, and it indicates the event at the exit of logic gates (top or mediator), which occurs as a result of a logical combination of the input events. It contains a description of the event. The circle and diamond are the most frequently used of all symbols for basic events. The circle indicates the basic initiating event that requires no further development. It represents the completion of the trees at this point. The diamond indicates an event that has not been developed to its own causes due to lack of necessary information, or the low significance of its consequences, or the avoidance of parallel analysis.

Logical symbols in the fault tree signify mutual conditionality and correlation of events of the lower and higher level. Thus, for example, "OR" logic gate produces output event if one or more of the input events occur. In contrast, "AND" logic gate produces output event if all of the input events occur. In addition to the symbols of logic gates listed in Table 1, there are a number of others less-used symbols.

Table 1. Explanations of the constituent symbols of the fault tree

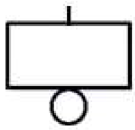
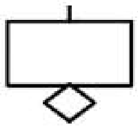
Name of the symbol	Graphic representation
Top or mediator event	
Primary – basic fault event	
Secondary – basic fault event (Undeveloped event)	
OR gate	
AND gate	
Transfer in	
Transfer out	

Symbols for transfer in the form of triangles, with the identification mark in the form of letters within them, enable forming of the complex fault trees in the form of the parent tree and a number of sub-trees. In addition, if there are identical parts of the fault tree, using these symbols, unnecessary repetition of the same parts of the tree is avoided, and therefore, achieves the savings in space and the visibility of the tree is also better.

There is a requirement to the analyst who, before start forming fault tree, should very well research the considered system from the standpoint of structure, operation mode and the relationship between the components. Only a full understanding of operation mode of the system and its elements, as well as knowledge of their mutual relationships, allows the implementation of logical analysis that defines the necessary and sufficient conditions for the realization of the top event.

In the formation phase of the observed object's fault tree, the modified symbols for primary basic event (circle) and secondary basic event (diamond) are used in this paper, for practical reasons, and are shown in Table 2 [11].

Table 2. The symbols of the primary and secondary basic events

Name of the symbol	Graphic representation
Primary – basic fault event	
Secondary – basic fault event (Undeveloped event)	

The considered process of obtaining granules for the production of plastic masses by chemical polymerization, which is carried out in a system with a VK-pipe, is shown in Figure 1. The produced polymer may have different properties, depending on the values of variables in the technological process. For every phase of the polymerization, whether it is pre-polymerization, polymerization or post-polymerization, the values of the variables are prescribed, in order to obtain a product with the required properties.

In the analysis of irregularities in the production process, which can result in variations of the final product's characteristics, for the top event in the fault tree "The process of obtaining sintered polyamide 6 is irregular" was adopted, that is shown in Figure 2. In developing the top event, the structural approach is used. As the polymerization process consists of three phases, it is evident that irregularities in any one of the three phases of the events, leads to the top event.

By the deductive analysis of these events it was made the completion of the fault tree structure by causes that contribute to them in the form of fault tree branches. Development of the mediator event "Irregular process of pre-polymerization" was carried out in the scope of an independent sub-tree that is shown in Figure 3. The mediator event "Irregular process of polymerization" has been developed up to the level of the basic events within the parent fault tree (Figure 2). Finally, the development of mediator events "Irregular process of post-polymerization" is presented as an independent sub-tree in Figure 4. In this way, in addition to identifying and recording potential causes of bad characteristics of the final product of polymerization of plastic masses, the causal link between basic events, intermediate and top event was established.

Development of the top event in the tree "Process of obtaining sintered polyamide 6 is irregular" to basic primary and secondary events provides quality and quantity analyses of the fault tree of the considered object.

4. QUALITATIVE ANALYSIS OF THE FAULT TREE

Qualitative analysis of the fault tree means [10]: estimation of the minimal cut sets, ranking elements regarding their importance and analysis of the common cause (common mode) failures.

Cross-section is a set of system events which cause the system top event to occur in the fault tree. That cross-section of an event on the fault tree is called disjunctive set because it leads to failure of the observed object. Minimal cross-section is set of events, which cannot be further reduced and, which also cause the top event to occur. Similarly, a set of minimum cross-section of the event is such a set consisting of all minimal cross-sections of events for one tree that cannot be reduced [11,13].

To determine the set of minimum cross-sections of events it is required that all events in the tree denote with Boolean variables. It is common that top event is marked with T, mediator events with G_i (i = 1, 2, ...), basic events with E_j (j = 1, 2, ...) or P_n (n = 1, 2, ...) and S_m (m = 1, 2, ...) (primary and secondary events).

In order to perform qualitative analysis of a tree of the irregular process for obtaining sintered polyamide 6 (Figures 2, 3 and 4), all events on the fault tree are marked with Boolean variables and represented in Figure 5. These figures show that fault tree with the top event "Process of obtaining polyamide 6 is irregular" has 10 mediator and 23 basic events (10 primary and 13 secondary events).

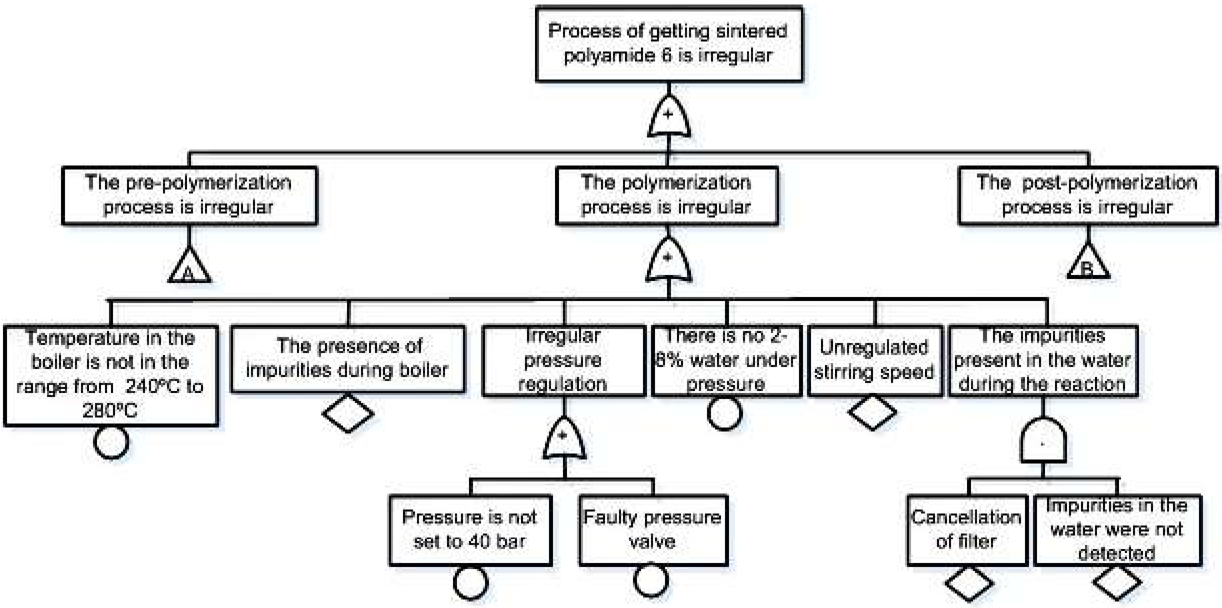


Figure 2. Fault tree for the top event "Process of obtaining sintered polyamide 6 is irregular"

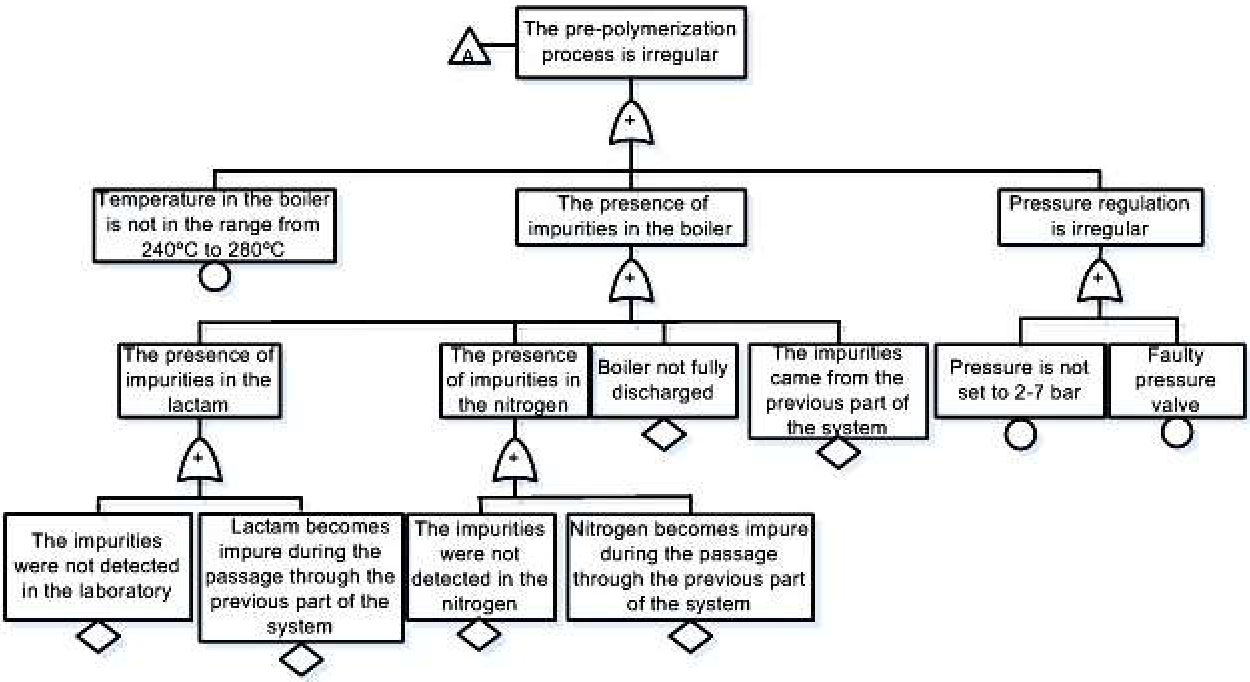


Figure 3. Independently sub-tree of the irregular pre-polymerization process

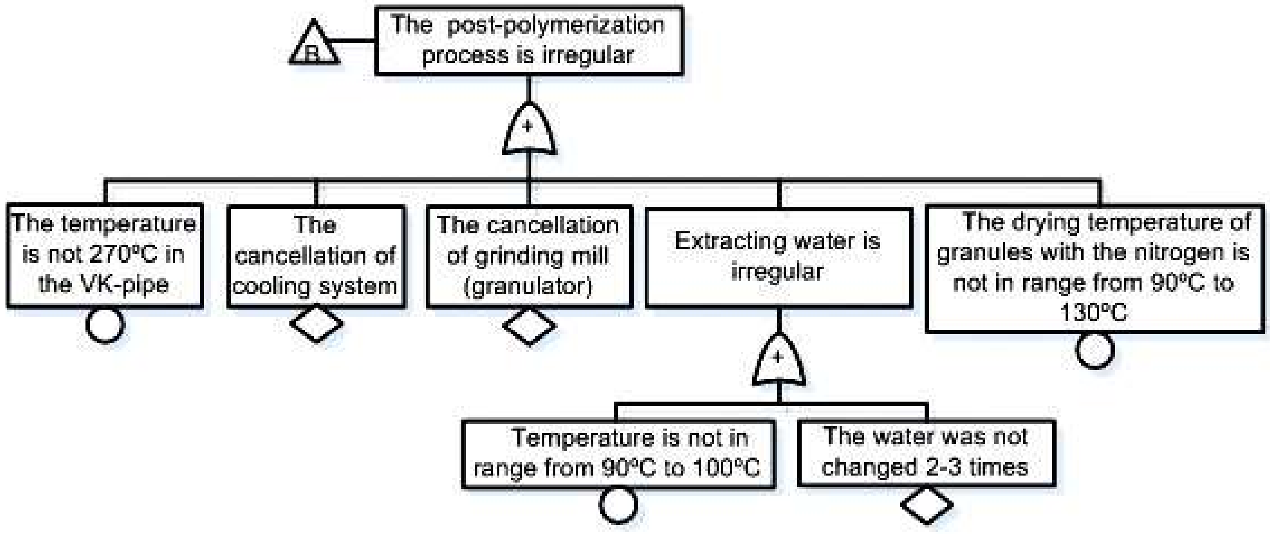


Figure 4.Independently sub-tree of the irregular process of post-polymerization

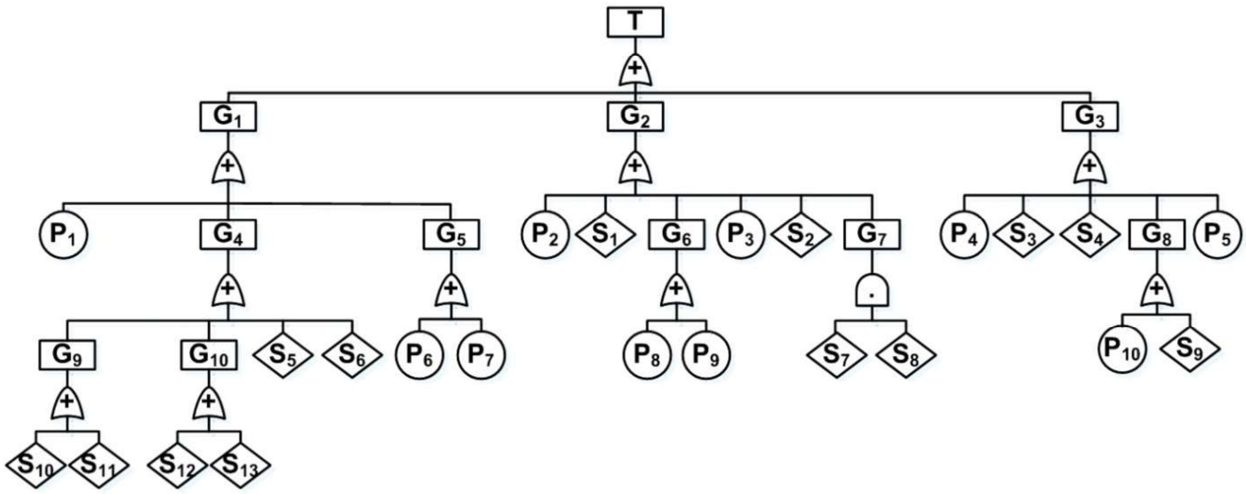


Figure 5. Developing the top event "Process of obtaining sintered polyamide 6 is irregular" through mediator events up to the basic events

Proceeding of determination of minimal cross-sections has three steps [10,11,14]:

1. Forming logic equations for each logic circuit in the fault tree. Each logic circuit has one corresponding equation. For the fault tree on Figure 5 it is obtained:

$$T_1=G_1+G_2+G_3,$$
$$G_1=P_1+G_4+G_5,$$
$$G_4=G_9+G_{10}+S_5+S_6,$$
$$G_9=S_{10}+S_{11},$$
$$G_{10}=S_{12}+S_{13},$$
$$G_5=P_6+P_7 \qquad (1)$$
$$G_2=P_2+S_1+G_6+P_3+S_2+G_7,$$
$$G_6=P_8+P_9,$$
$$G_7=S_7\cdot S_8,$$
$$G_3=P_4+S_3+S_4+G_8+P_5,$$
$$G_8=P_{10}+S_9.$$

2. Expressing top event in term of basic event by replacing some mediator events with corresponding expressions:

$$T=P_1+S_{10}+S_{11}+S_{12}+S_{13}+S_5+S_6+ +P_6+P_7+P_2+S_1+P_8+P_9+P_3+ +S_2+S_7\cdot S_8+P_4+S_3+S_4+P_{10}+S_9+P_5. \qquad (2)$$

3. Reduction of obtained expression by using rules of Boolean algebra. For the coherent fault trees (fault trees where does not exist special logic circuits which use negation and two basic opposite events) it is enough to use only idempotency law ($P{\cdot}P = P + P = P$) and law of absorption ($P + P \cdot Q = P{\cdot}(P + Q)$) [13,14]. In mentioned example expression obtained after the second step can't be further reduced.
Using described procedure, all cut sets of a fault tree are obtained after the second step, and minimal cut sets are obtained after the third step. In other words result of third step is minimal disjunctive normal form.
Analysis of the common cause is very important part of the qualitative analysis of the fault tree. It is used for analysing behaviour of the system exposed to special conditions and secondary causes [11].
Special condition is characteristic which connects closely several primary events (for example, elements of the same producer).
Secondary cause is event which may contribute occurrences of several basic events in the fault tree.
In the irregular procedures for obtaining sintered polyamide 6, all secondary causes that lead to basic events, can be classified into three categories:

A –irregular pre-polymerization process,
B - irregularpolymerization process and
C - irregular post-polymerization process.

Based on fulfilled categorization of the basic events of the fault tree of the irregular process of obtaining sintered polyamide 6,according to secondary causes, set of events, which cause the system top event to occur, could be written in a form:

$$T{:}\{9 \text{ categ.}A,\ 7 \text{ categ.}B,\ 6 \text{ categ.}C\}. \qquad (3)$$

Analysis of the common cause means ranking of influences of particular secondary causes (*A*, *B*, *C*) to the top event *T*. In that way relative participation (*RP*) of each of the four causes is obtained:

1. $RP_A = 0{,}4091$,
2. $RP_B = 0{,}3182$,
3. $RP_C = 0{,}2727$,

where:

$$\sum_{i=A}^{C} RU_i = 1.$$

The relative contribution of each of four secondary causes of basic events is shown on Figure 6.
Considering obtained results, according to Figure 6, it can be seen that the irregular process of obtaining sintered polyamide 6 is most affected by inadequate pre-polymerization process with 41%.

Maintenance and control of the required temperature and pressure, first of all, affect the irregularity of the pre-polymerization process. Furthermore, it is important that the boiler, before each polymerization process, is entirely drained, and the reagents that are added in the boiler are completely clean.

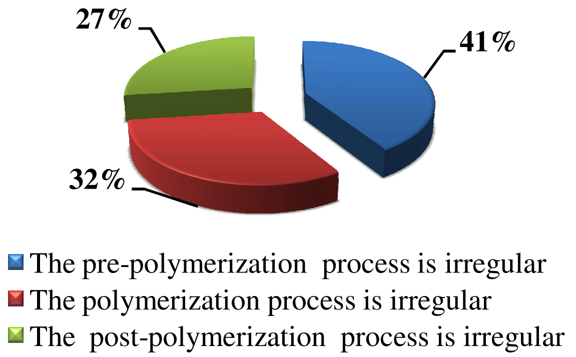


Figure 6. Participation of the basic events secondary causes expressed in percentages

In order to obtain fully regular process, it is necessary to control the operation of the entire system for polymerization, and particularly the maintenance of temperature and pressure within the set limits. Only in this way can be achieved obtaining a desired quality of polyamide 6.

5. CONCLUSION

Fault tree analysis, based on the design of fault tree's scheme, is applied in many areas of technics. FTA method can be also applied within the field of chemical technology that is shown in this particular example.
For the application of this model, obtained based on the deduction, it is required a complete knowledge of the analysed system, and its constituent elements, their technological connections, as well as relevant data.
This paper shows that the complex technological process can be described by simple symbols and logical relationships between events, which significantly simplifies the practical application, especially in the case of the analysis, control and minimize the losses within the system.
The application of FTA method enables a detailed analysis of the considered technological system from the aspect of occurrence of failures during operation.
Failure analysis during operation is especially important in technological systems because their failures lead to heavy financial losses and severe consequences for humans and the environment. Systematic analysis of potential failures in the operation of technological systems is essential for

the planning of maintenance. This may have a crucial impact on their effectiveness.

The fault tree of the considered technological system presents a convenient means used for illustration of the advantages of proposed solution, in regard to other solutions, i.e., it is material for argument discussion. If designed system contains errors, fault tree can help in finding weak points, and it could show how these points lead to the unwanted events. In a properly designed system, all potential causes of system failure can be predicted by using the fault tree.

By the formation of the fault tree for the sufficiently general top event can be recorded most of the potential failure modes of the technological system's elements, which can be used as the basis for Failure mode and effects analysis - FMEA.

6. LITERATURE

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