

# Risk Assessment of Batch Sulfonation Process Using Fault Tree and Bowtie

Soham Dalwadi<sup>1</sup>, Chirag Parmar<sup>2</sup>, Siddharth Modi<sup>1,\*</sup>

## Abstract

The current study looks into the batch reactor's sulfonation reaction's safety features. Acetanilide, thionyl chloride, and chlorosulphonic acid are used in the sulfonation reaction under consideration to generate acetyl sulphonyl chloride as well as by-products such HCl and SO<sub>x</sub>. Steam starts the process, which is exothermic in nature. Water is then chilled to maintain a temperature of 50°C and a pressure of one atmosphere. The Hazard and Operability (HAZOP) analysis was carried out initially, followed by Fault Tree Analysis (FTA), Event Tree Analysis (ETA), and Bow-Tie Analysis (BTA), in light of the safety concerns related to the reaction in question. The techniques used assisted in identifying the inherent dangers associated with this intricate procedure. Methodically examining the process parameters, the HAZOP analysis looks for variations in the process that could result in risks like uncontrollable reactions or operational failures. The findings of the HAZOP study were incorporated into the fault tree, event tree and bowtie by considering reactor failure chances as the top event. The proposed fault tree had 17 intermediate events, and 30 basic events. Critical parameters such as temperature and pressure have a significant impact on the process. For example, temperatures above 150°C have the potential to cause thermal runaway and concurrent overpressure events. The current work contributed to ensuring the efficiency, dependability, and safety of the sulfonation reactions in batch processes by employing a mix of risk assessment tools.

**Keywords:** Risk assessment, sulfonation reaction, hazard operability, fault tree analysis, bowtie analysis

## INTRODUCTION

Chemical process safety must be guaranteed in order to shield workers and the environment from potential dangers. The batch sulfonation process is an industrially important critical operation that involves chemicals like acetanilide, thionyl chloride, and Chloro-Sulfonic Acid (CSA). This batch process is crucial for various industrial sectors including pharmaceuticals, dyes, and surfactants which demands meticulous attention to prevent accidents and mitigate risks effectively.

### \*Author for Correspondence

Siddharth Modi  
E-mail: [siddharth.modi.ch@ddu.ac.in](mailto:siddharth.modi.ch@ddu.ac.in)

<sup>1</sup>Research Scholar Department of Chemical Engineering, Dharmsinh Desai University, Nadiad, India

<sup>1</sup>Assistant Professor, Department of Chemical Engineering, Dharmsinh Desai University, Nadiad, India

<sup>2</sup>Research Scholar Plant and Process Safety Division, Bodal Chemicals Ltd, Vatva, India

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The batch sulfonation process mentioned here initiates at 50°C, with steam circulation through coils to trigger the reaction. Manual addition of acetanilide ensures precise dosing and uniform mixing while chilling water circulation serves to control the exothermic reaction and prevent overheating. The by-products such as HCl and SO<sub>x</sub> are directed to a scrubber system, ensuring environmental compliance. Subsequently, the reaction mass undergoes transfer to a dumping tank for further processing and purification.

To evaluate and enhance safety in this intricate process, various tools including Hazard and

Operability (HAZOP) Study, fault tree, event tree, and bowtie have been employed. Each analysis method offers unique insights into potential hazards, causes, consequences, preventive measures, and mitigation strategies, collectively forming a robust safety framework [1].

### HAZOP Study

The HAZOP study examines the deviation of critical parameters such as temperature, pressure, concentration, level, and flow rate with respect to their causes, effects, and necessary preventive and mitigating actions. In the present work, a detailed HAZOP study was conducted for each parameter and based on the study the most critical one affecting the process significantly i.e. temperature and pressure are presented here. The batch reactor was considered as a node for which temperature and pressure deviations and their corresponding causes, consequences, and actions are presented in Table 1 and Table 2 respectively. Through this analysis, preventive measures including the implementation of safety guards and instruments are identified to mitigate risks effectively [2].

**Table 1.** Temperature deviation in HAZOP study for batch sulfonation.

Parameter	Guide word	Possible causes	Possible consequences	Safeguard	Action required
Temperature	More	Less chilling water supply; Coolant line blocked; Coolant pump failure; More quantity of CSA; Non-uniform agitation operation; Presence of contamination	Exothermic reaction Temperature rise beyond control; Thermal decomposition of chemicals; Pressure build-up in the reactor; Possible thermal runaway.	Proper sizing of vent is necessary; Multiple temperature measurement devices; Proper sizing of Safety valve; Pressure gauges are required; Requirement of sight glass; Requirement of flow meters in coolant line.	Standard Operating Procedure to be followed; Frequent cleaning and maintenance of the coolant system and agitator system is a must; Frequent Calibration of transmitters.
	Less	More coolant flow than desired; Inappropriate amount and sequence of chemical dosing.	Deterioration in the product quality; No extreme hazard.	Continuous temperature measurement; Requirement of flow meters in coolant line.	Standard Operating Procedure to be followed.

**Table 2.** Pressure deviation in HAZOP study for batch sulfonation.

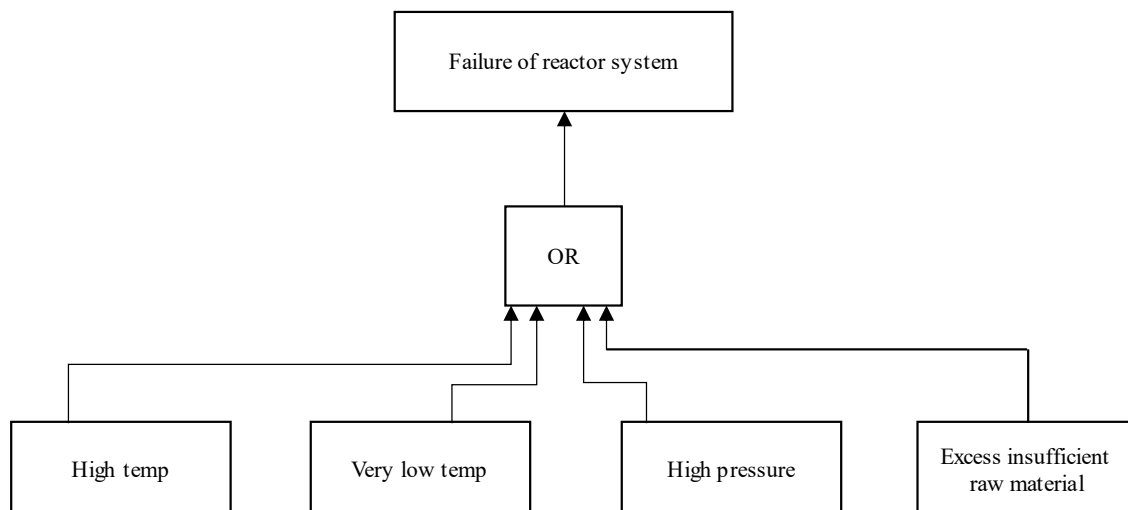
Parameter	Guide word	Possible causes	Possible consequences	Safeguard	Action required
Pressure	More	More quantity of CSA leads to process upset; Entry of water or other contaminant in the reactor; No scrubbing of HCl & SO <sub>x</sub> leading to accumulation of vapor in the reactor;	Uncontrolled rise of pressure and temperature; Exothermic process;Threat to vessel mechanical integrity.	Appropriate Vent to the reactor; Pressure gauges on the reactor;Temperature monitoring is equally important; Proper sizing of safety valve.	Standard Operating Procedure to be followed;Frequent cleaning and maintenance is a must;Frequent Calibration of transmitters.
	Less	Possible leakage in the system; Inappropriate reaction condition; Reaction does not take place possibly due to contaminants.Wrong addition of chemicals leading to no reaction.	Deterioration in the product quality;No extreme hazard.	Continuous pressure and temperature measurement;Check for any leakages in the system.	Standard Operating Procedure to be followed.

The HAZOP study suggest to have detail investigation on high temperature and pressure chances qualitatively. The coolant system, blockages and leakages in the system, possible thermal runaway must be investigated using other risk assessment tools fault tree, event tree and bowtie [3].

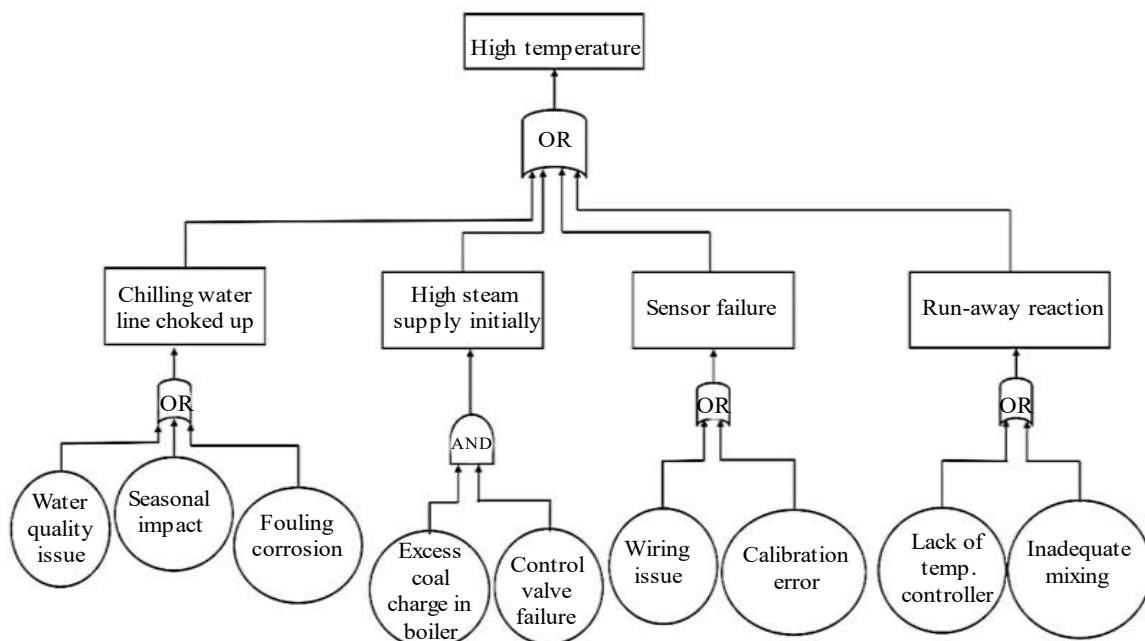
### Fault Tree Analysis

A systematic graphical technique called fault tree analysis (FTA) is used to find and examine possible failure modes in complicated systems.

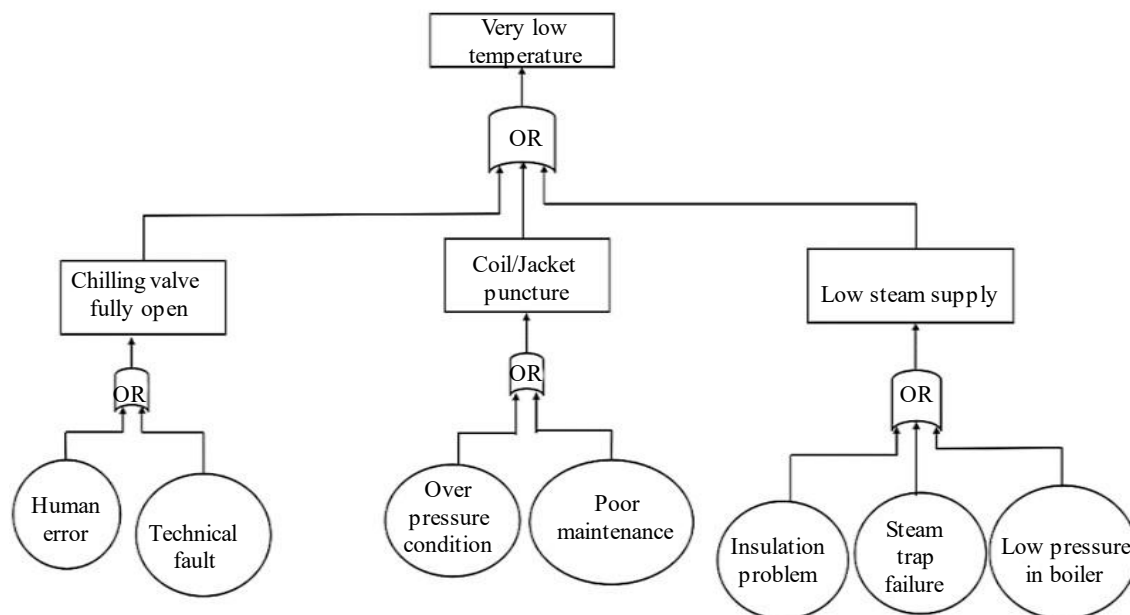
FTA begins by identifying the top event of interest, such as reactor system failure in the batch sulfonation process, and then systematically breaks down this event into its contributing intermediate and basic events. Each event is represented graphically in a tree-like structure, with logic gates indicating how events are related and influence one another. By analysing the fault tree, we can identify critical failure modes, assess their probabilities, and develop targeted preventive measures and mitigation strategies to enhance the safety and reliability of the system [4].



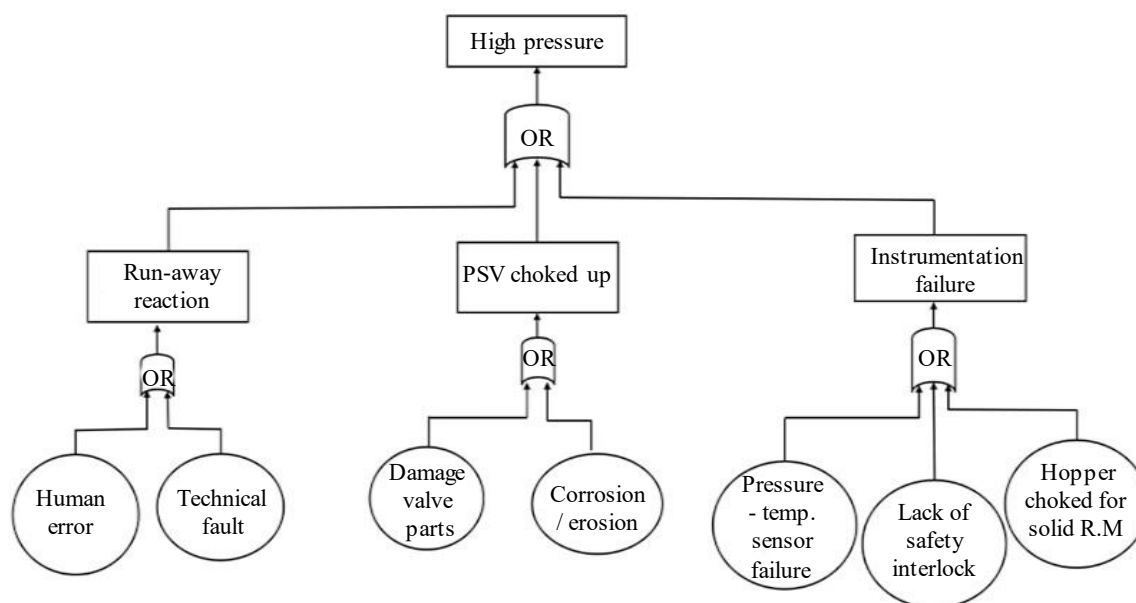
**Figure 1.** Fault tree for reactor failure as top event



**Figure 2.** Fault tree for high temperature as first intermediate event



**Figure 3.** Fault Tree of low temperature as second intermediate event

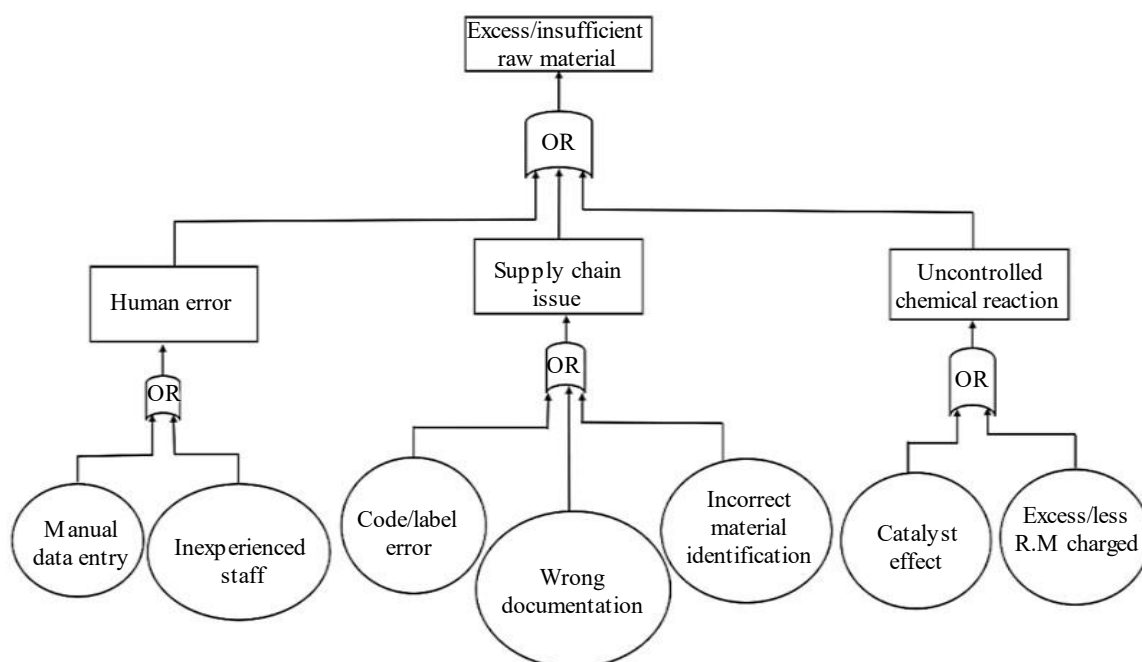


**Figure 4.** Fault Tree of high pressure as third intermediate event

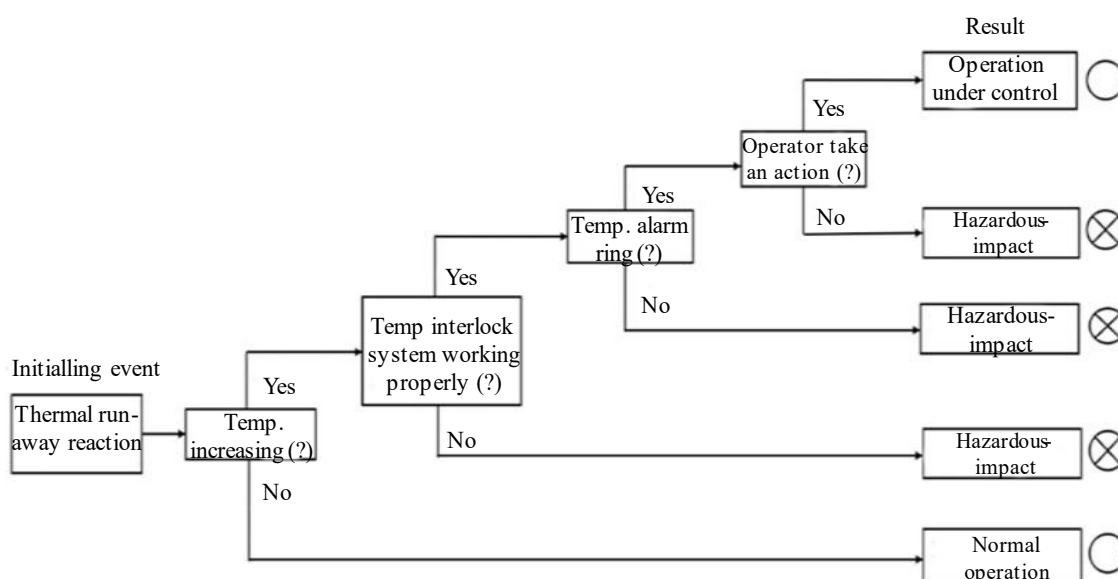
In the present work, the reactor failure is considered as undesired top event and the tree network is shown in Figure 1. The four intermediate events associated with the undesired reactor failure system are very high temperature (shown in Figure 2), extreme low temperature (in Figure 3), high pressure (in Figure 4) and raw material variation (in Figure 5). [5]

**Event Tree Analysis**

The Event Tree Analysis (ETA) delineates potential sequences of events stemming from the initial event. By assessing the temperature trends and the efficacy of temperature interlock systems and alarms, the ETA analysis provides a structured approach to anticipate and prevent hazardous impacts, emphasizing the importance of operator intervention in averting catastrophic events. The event tree for initiating event leading to thermal runaway is presented in FIGURE 6. The events sequence leading to hazardous impact are marked as cross inside the circle in the same Figure [6].



**Figure 5.** Fault tree of raw material variation as the fourth intermediate event

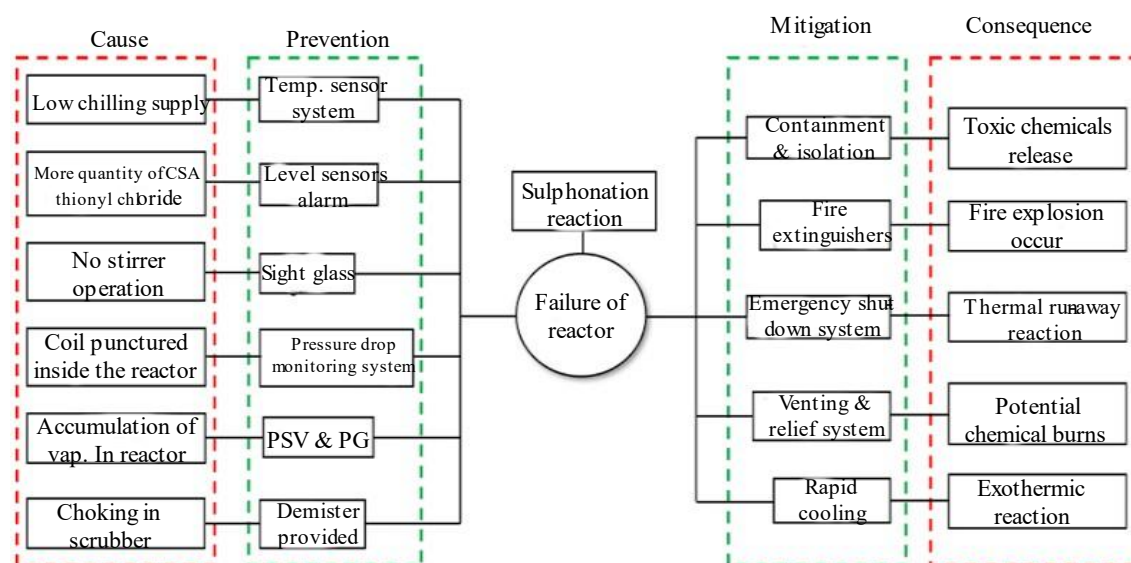


**Figure 6.** Event tree for initiating event leading to thermal run-away

### Bowtie Analysis

The Bowtie Analysis (BTA) serves as a visual tool to identify causes, consequences, preventive measures, and mitigation strategies associated with the failure of the reactor system. By pinpointing potential causes such as low chilling water supply or coil puncture, and proposing preventive instruments like temperature sensors and alarms, this analysis offers a comprehensive approach to risk management [7].

In the present work, the fault tree proposed in Figure 1 to Figure 5 and the event tree proposed in Figure 6 are combined to develop a bowtie for the sulfonation reactor failure system with cause and prevention on one side of the tie and consequence and mitigation on the other side of the tie as depicted in Figure 7. [8].



**Figure 7.** Bowtie diagram for batch reactor.

The overall risk management plan suggests low chilling supply, more CSA quantity, poor agitation, rupture of the coil, accumulation of vapor, and choking inside the scrubber are possible causes for which temperature sensors, level sensors, sight glass, pressure monitoring, safety valve, and demister pads as part of preventive measures. The toxic chemical release, fire chances, thermal run away chances and uncontrolled exothermic reaction are potential consequences of the discussed causes for which contamination isolation, fire extinguishers, emergency shut down system, venting and relief system and rapid emergency cooling system are the proposed mitigating actions for the sulfonation reactor system failure. [9, 10].

## CONCLUSION

As we delve deeper into the safety analysis of the batch sulfonation process, it becomes evident that a multi-faceted approach encompassing various analytical techniques like HAZOP, FTA, ETA, and BTA is indispensable for mitigating risks effectively. By implementing preventive measures and mitigation strategies proposed through the above-mentioned tools, we aim to bolster safety protocols, ensuring a secure operational environment conducive to sustainable industrial practices.

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