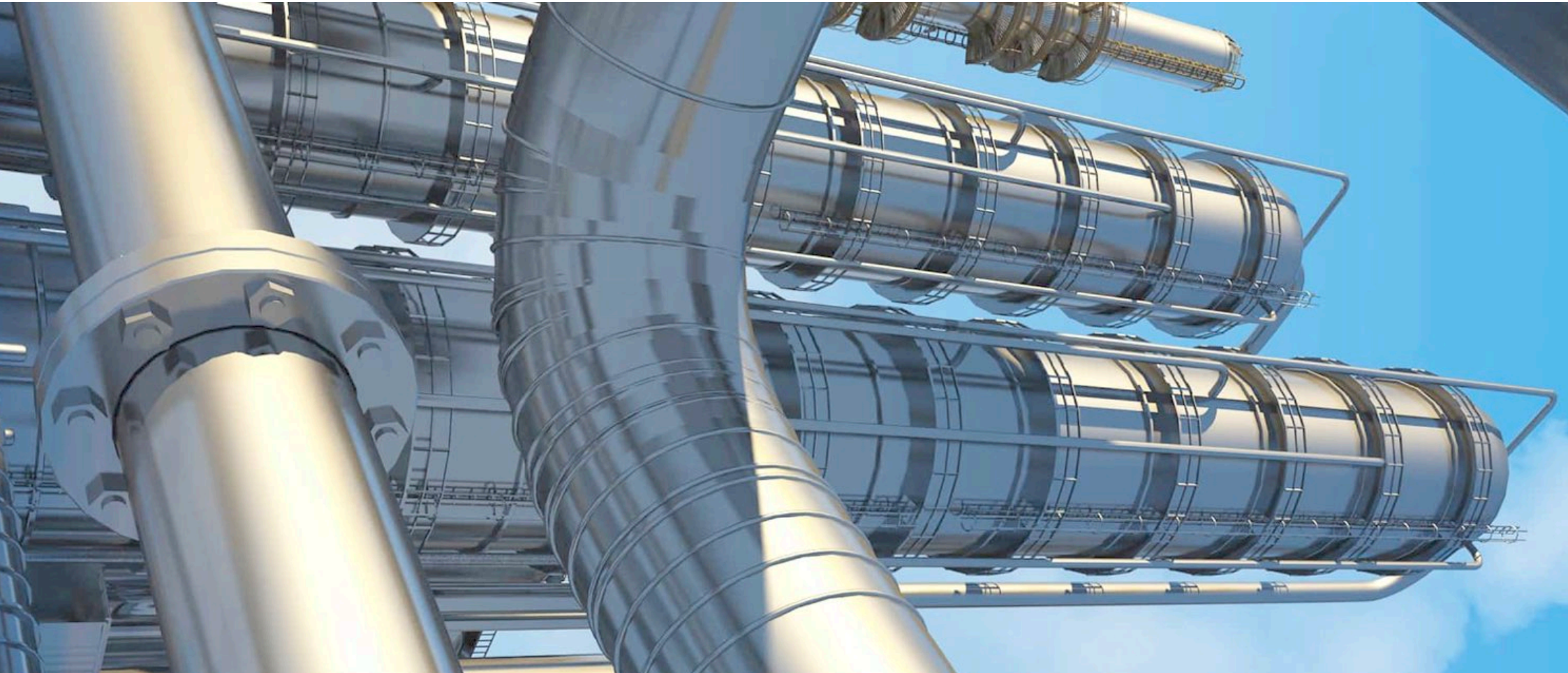
Proprietary process technology

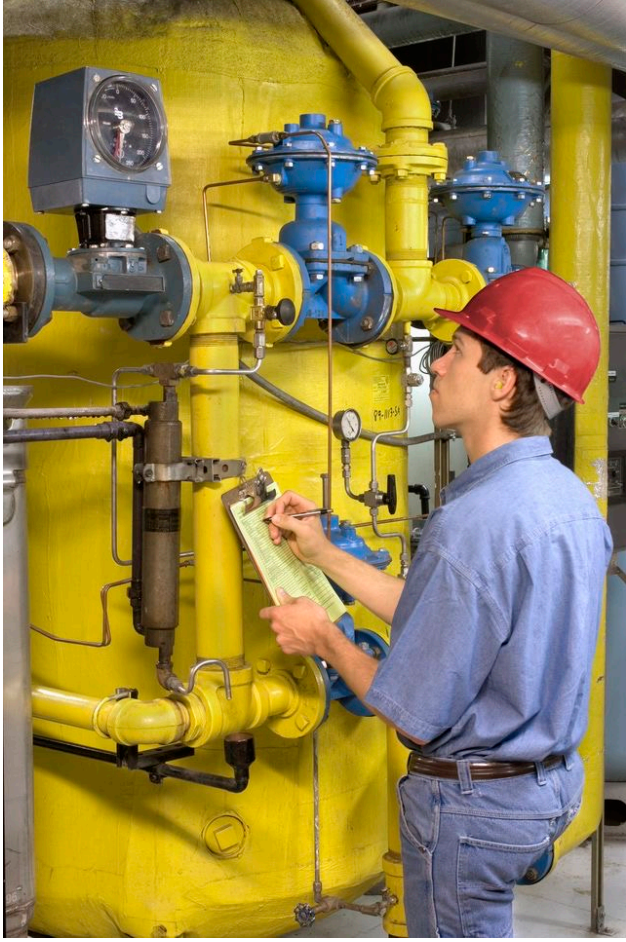
ADVANCED

ISP

paraformaldehyde isobutene

Gas-phase Synthesis at high pressure





工业气相合成异戊烯醇简述

Description for Industrislly Synthetic Isoprenol Process in Gas-phase

异戊烯醇，3甲基-3丁基-1醇，CAS No.763-32-6， 是一种萜醇主要用于加工聚羧酸水泥减水剂的基本原料。

异戊烯醇是依普林斯反应将醛类及烯类高压，气相加成合成生成。显著不同于市场其他异戊烯醇生产工艺。

Isoprenol, 3-methyl-3-buten-1-ol, CAS No. 763-32-6, is a kind of Terpene alcohol, mainly using primary material to process polycarboxylic acid cement water-reducing additive. Isoprenol is synthesized by aldehydes and olefins under high pressure in gas phase, called as Prins Reaction. It is quite different with other process in Market.

气相合成工艺与国际市场其他溶液，容剂及催化剂工艺比较

comparative between gas-phase process and solvent, solution and catalytic process in the in the international market

1. 溶液法工艺：

Synthesize isoprenol in solution process

早在1970年，飞利浦公司使用甲醛溶液和甲醇生产异戊烯醇。甲醇和甲醛摩尔比为10:1；反应温度：275℃-300℃；压力138Kg/cm².异戊烯醇收率80%。该工艺缺点：产品中杂质多，副产品多；高温；高压。

In the end of 1970 Phillips Petroleum Company synthesized isoprenol by using formaldehyde in aqueous solution and solution with methanol. Methanol in a molar relationship 10 times bigger to FA, to 275-300° C and 1,38*10⁷Pa with a ISP yield of 80%. Disadvantages were for the product purity and for the pressure and operating temperature conditions of the process also high, as well as many by-products.

2. 使用有机溶剂苯合成异戊烯醇工艺：

Synthesize isoprenol by using organic solvents like benzene

异丁烯和甲醛摩尔比大于11:1；温度200℃；批次反应反应时间6小时。异戊烯醇收率：85%。该工艺缺点：产品纯度低，反应收率低。

The use of organic solvents, like for example the benzene, allows to realize the process, with ISB in a molar relationship as regards the FA of 11:1, at 200° C for 6 hours with a yield in ISP of 85%. The use of organic solvents brings some disadvantages for the product purity and the reaction yield low.

3. BASF工艺：

Synthesize isoprenol by BASF

50%水溶多聚甲醛，氮气加压至250Kg/cm²;温度：250℃，异丁烯和甲醛摩尔比为10:1；批次操作反应时间：1小时。异戊烯醇收率达96%。该工艺缺点：生成大量污水；高温高压。工业化和投资均不可行。

The aqueous PFA used at 50% and the use of nitrogen to pressurize till a total pressure of 2,5*10⁷Pa. The reaction takes place at 250° C with a molar relationship ISB:FA of 10:1 and with 1 hour of residence time.

The yield in ISP is 96%, the major part of the sewage to be treated around 10 times more than the current process as well as the high pressure and temperature in the process.

The difficult operating conditions (high temperature and pressure), don't allow to the process to get importance from an industrial and financial point of view.

4. 催化剂选择合成工艺：

The process for selective synthesis of catalyst:

非均质催化剂：

Catalyst in Heterogeneous

催化剂采用金属羰基化合物，甲醛或聚甲醛溶在有机溶剂中；温度：200℃；反应周期1小时。该工艺缺点：在使用WO3金属氧化物为催化剂，甲醛转化率70%。问题是该种催化剂对异戊烯醇选择性较低

Metallic carbonyls and oxides were selected to use of FA or PFA solutions in organic solvents at a temperature of 200° C for one hour. With these catalytic systems it obtained low yields of ISP (till 70% of FA conversion, with catalyst based on WO_3). The problem in this system was the scarce selectivity to ISP synthesis reaction.

均质催化剂：

Catalyst in homogeneous

结合到离子交换树脂的金属氯化物Sncl和Zncl. 反应条件温和，温度<100℃，常压，但收率低。尽管在水溶液状态下可提高催化剂活性，异戊烯醇的选择性低。

Sncl和Zncl 结合到离子交换树脂上。可以获得四烷基铵，锌,硅和沸石复合物例如，催化剂ZSM-5 和FeMCM-22. 异戊烯醇合成的选择性低，收率低。

催化反应另一个问题是异戊烯醇由催化剂中及由未反应物中分离困难。工艺收率低，反应选择性低。催化剂昂贵。

The use of Catalyst in homogeneous like the metallic chlorides (for example $SnCl_4$ and $ZnCl_2$) bound to a resin with ionic exchange with soft conditions (temperatures <100° C and room pressure) brings to low yields, especially in presence of water. In anhydrous conditions, these catalytic systems have a higher activity but a low ISP selectivity.

The use of $SnCl_4$, $ZnCl_2$ bound to a resin with ionic exchange has been experimented and it contains Tetraalkylammonium, complexes of tin, silica and zeolite like ZSM-5 and FeMCM-22. Also in this case the scarce selectivity of the catalysts for the ISP synthesis, brings to a scarce process yield.

The above mentioned catalytic processes show some inconveniences bound to the separation of the ISP from the other reaction mixture components. In particular the reaction mixture，including catalyst is hardly separable. It was together with the scarce processes yield due to the low reaction selectivity, making the processes expensive and not so exploitable.

气相合成工艺装置特色

characteristics for synthesis process in gas-phase

原料在市场上普遍，易得。

The raw materials are easy to find and to procure in the market

工艺原料及化学品：

Raw materials and chemicals in process

1 多聚甲醛

Paraformaldehyde

2 异丁烯

Isobutene

3 苛性钠

Caustic soda

生产操作简单。工艺中仅两个控制参数：温度，压力；连续化工艺，连续回收未反应物，优化了反应物消耗。

Production process is very simple. In process there are only two parameters such as temperature and pressure to be controlled, continuous process, continuous recovery for un- reacted materials, optimizing the consumption of reaction materials.

装置简单，设备少（装置中反应器，泵，塔，换热器，储罐…总计124台）

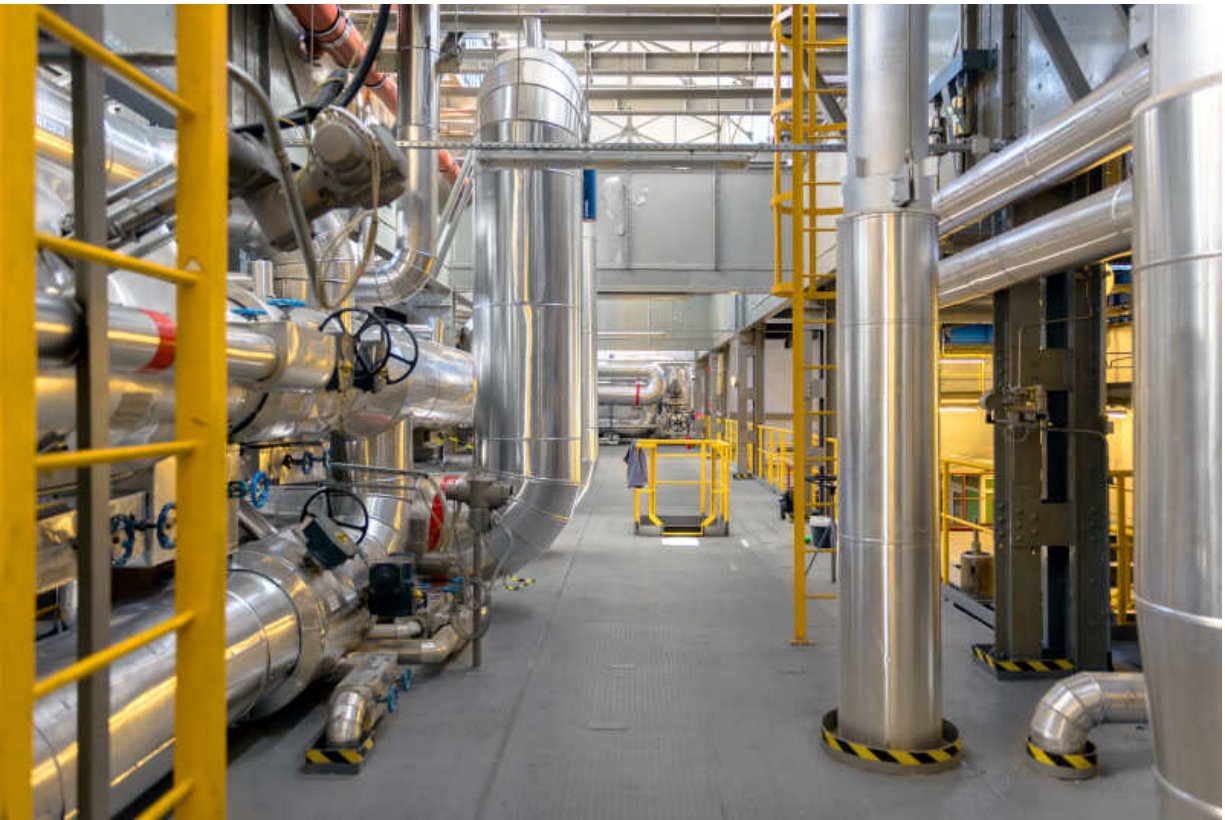
The plant composed is also simple with less equipment installed (reactors, pumps, towers, heat exchangers and tanks…total 124 sets)

连续化生产

Continuous process

投资少

Investment less



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气相合成工艺特色

characteristics for synthesis process in gas-phase

因气相合成工艺无溶剂，无水溶液，无催化剂，不存在异戊烯醇由溶剂或由催化剂中分离操作，获得无污染高纯度的异戊烯醇。

As are no solvent, catalysts and water in gas-phase process it does not need to separate isoprenol from solvent, catalyst or water so that it can obtain isoprenol with high purity and no pollution.

因无催化剂，避免异戊烯醇由催化剂中分离。不使用昂贵催化剂大大降低工艺成本。

As no catalyst existed in process separation between isoprenol and catalyst does not need. The cost of process is tremendously reduced because there is no catalyst application.

异戊烯醇的选择性高。

Selectivity high for isoprenol

对多聚甲醛收率为97%，对异丁烯收率为90%

97% yield to Paraformaldehyde and 90% to Isobutene

无污染物排放

No pollution discharged from plant

工艺中无催化剂，无溶剂，无三废污染物排放是绿色环保工艺

There is no catalyst, no solvent, no solid wastes discharged from process, protecting environment.

占地小（装置总占地：1万平方米）

Small land occupation (plant area: ten thousand square meters)

投资少

由于原料在市场上易得，连续化工艺简单，设备台数少及环保无三废排放装置总投资少

As the raw materials are easy to procure in the market and continuous process is simple and number of equipment installed is less and no pollution discharged from plant so total investment in plant is also extremely reduced.

异戊烯醇产品标准：

Specification of isoprenol product

1 纯度：>98%

Purity: >98%

2 水分： <0.1%

Water: <0.1%

使用该技术的装置运行多年实践证明装置安全可靠没有任何工艺，设备事故。产品质量稳定始终达到国际标准。

Operation practice strong demonstrated that the plant is safety, reliable, without any accidents occurred in process and equipment. The quality of product always is stable, reaching the international standard

异戊烯醇技术及工业化装置获得国家发明专利 CN 112430178 A .具有自主知识产权。

The technology of isoprenol and its industrial plant also obtained an invention patent in State CN 112430178 A, having independent intellectual property rights.

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