

Review

The Production of Isophorone

Timm Ruther ¹, Marc-André Müller ², Werner Bonrath ² and Matthias Eisenacher ^{1,*} 
¹ Circular Transformation Lab Cologne, TH Köln-University of Applied Sciences, 50678 Köln, Germany

² DSM Nutritional Products, 4303 Kaiseraugst, Switzerland

* Correspondence: matthias.eisenacher@th-koeln.de

Abstract: Isophorone is a technically important compound used as a high-boiling-point solvent for coatings, adhesives, etc., and it is used as a starting material for various valuable compounds, including isophorone diisocyanate, a precursor for polyurethanes. For over 80 years, isophorone has been synthesized via base-catalyzed self-condensation of acetone. This reaction has a complex reaction mechanism with numerous possible reaction steps including the formation of isophorone, triacetone dialcohol, and ketonic resins. This review provides an overview of the different production processes of isophorone in liquid- and vapor-phase and reviews the literature-reported selectivity toward isophorone achieved using different reaction parameters and catalysts.

Keywords: catalysis; self-condensation; acetone; isophorone; base-catalyzed

1. Introduction

In celebration of the 60th anniversary of the isophorone (IP) production at the Hilbernia AG production site in Herne (Germany), which today belongs to the Evonik AG [1,2], this review provides a detailed overview of the current state of science of the product IP, its applications, its production methods, and reviews the catalytic research for its synthesis.

The technical importance of IP is justified by its use as a solvent and starting material for several chemical processes, and due to its possibility to be synthesized from acetone (Ace) [3–6]. Ace is the coproduct of the phenol production, the cumene process, with a global Ace production output of 7.8 Mio. tons in 2020 [7]. Although processes for the synthesis of IP from Ace were known before 1960, the major production of IP began in 1962 in West Germany [1,2,8]. Today, there are mainly two production methods for IP, a liquid- and a vapor-phase process. The estimated annual production volume exceeds 100,000 t/y [6] and the market is expected to further grow in the future [6,9,10].

2. Properties and Natural Occurrence

IP or α -isophorone, is an α,β -unsaturated carbonyl compound and a colorless-to-yellowish liquid [8,11–14] with a mint-like or camphor-like odor [13,14]. α -IP has two constitutional isomers (Table 1), β -isophorone and γ -isophorone [15]. Both are formed through the isomerization of α -IP [15–17]. Murphy et al. described that both the β - and γ -IP can be formed through the base-catalyzed isomerization of α -IP [15]. While the β -isomer is a common by-product of the isophorone synthesis [18], the γ -isomer is rarely reported in literature. This is supposedly due to the thermodynamical stability of the different isomers. Murphy et al. calculated that α -IP is the most stable and γ -IP the least stable of the three isomers [15]. Other options of the isomerization of α -IP are the acid-catalyzed isomerization to β -IP and the photocatalyzed isomerization to γ -IP [15–17].



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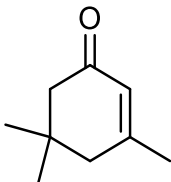
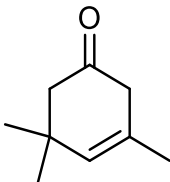
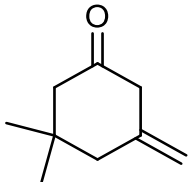
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Table 1. Physical properties of isophorone isomers [12,17,19,20].

Name	α -Isophorone	β -Isophorone	γ -Isophorone
structure			
CAS-number	78-59-1	471-01-2	72212-29-4
Molar mass	138.21 g/mol	138.21 g/mol	138.21 g/mol
Density (20 °C)	0.92 g/cm ³	0.91 g/cm ³	n/a
Melting point	−8.1 °C	−40.7 °C	n/a
Boiling point	215 °C	Decomposition	n/a

IP occurs naturally in a variety of different plants and animals [13,14,21]. Plants containing IP are, e.g., saffron crocus (*Crocus sativus*) [13,22,23], Mary's grass (*Hierochloe odorata*) [13], cranberry (*Vaccinium macrocarpon*) [13,14,24], and papaya (*Carica papaya*) [13,14]. Isophorone is formed, for example, as a pheromone, by different types of beetles, including the male specimens of *Homalinotus depressus* [25] and female specimens of *Ips typographus* [26,27], or as part of the defensive secretion of a grasshopper species [27]. Other isophorone sources are fly coal ash [28], Burley tobacco [13], black tea, Parmesan cheese, roast beef [13,14], and honey [29,30].

3. Applications and Synthesis

IP is mainly used as a high-boiling-point (Table 1) solvent [2,8,12–14,18,19] and as a starting material for the synthesis of different bulk and fine chemicals [3,8]. As solvent, it is used for solving polymers, oils, coatings, lacquer thinners, lacquers, adhesives, printer inks, paints, pesticides, etc. [2,12,18,31,32]. The few compounds that cannot be dissolved by isophorone include polyamides, polyurethanes, polyethylene, and polypropylene [8].

Additionally, IP is a starting material for the manufacturing of various important compounds, with examples shown in Figure 1 [3,8]. For example, ketoisophorone (P2) can be synthesized directly from IP but is often synthesized through the intermediate β -IP (P1) via oxidation [33–41]. Ketoisophorone (P2) is used to synthesize carotenoids [33,35], vitamin E [42,43], and flavoring agents [44–46] and is used as a fragrance additive [47]. Another product made from isophorone is 3,5-xylenol (P3), produced via catalytic isomerization [5,48–54]. 3,5-Xylenol is a starting material for various products, such as the disinfectants 4-chloroxylenol (P4) and 2,4-dichloroxylenol [8,50,54–56], resins [50], the insecticide Methiocarb [54], vitamin E [54], and dl- α -tocopherol acetate [8]. Through partial hydrogenation of isophorone, 3,3,5-trimethyl-1-cyclohexanon (P5) is produced [3,8,57–61], which can be used as a solvent [8] and for the synthesis of peroxide polymerization initiators [8,62]. 3,3,5-trimethyl-1-cyclohexanone (P5) can be oxidized by nitric acid to 2,4,4- and 2,2,4-trimethyladipic acid (P6), though the ratio depends heavily on the reaction conditions [63]. 2,2,4-Trimethyladipic acid (P6) is used to produce 2,2,4-trimethylhexane-1,6-diamine (P7) [3,64] and 2,2,4-trimethylhexane-1,6-diol (P8) [3]. 2,2,4-trimethylhexane-1,6-diamine (P7) can be used to synthesize polyamides [64] and 2,2,4-trimethylhexane-1,6-diol (P8) is used for polyester synthesis [3]. 3,3,5-Trimethyl-1-cyclohexanol (P9) is produced through the complete hydrogenation of isophorone [3,8]. 3,3,5-Trimethyl-1-cyclohexanol (P9) is used as a fragrance [8], as a fuel [65], and to produce vasodilator drugs [66–68], the combat agent EA 1511 (P10) [69,70] and the UV filter homosalate (P11) [71,72]. The most important products synthesized from isophorone are isophorone diamine (IPDA) (P12) and isophorone diisocyanate (IPDI) (P13) with a market value of over USD 600 million for IPDA in 2017 and a market value of over USD 700 million for IPDI in 2021 [8,73,74]. IP is

thereby converted in a three-reaction process to IPDA [3,8,75–80]. IPDA (P12) can either be used as a monomer for the polyamide synthesis [81] or phosgenated to yield IPDI (P13) as the product [64,76,82]. IPDI is used for the production of light-stable polyurethanes [8,64].

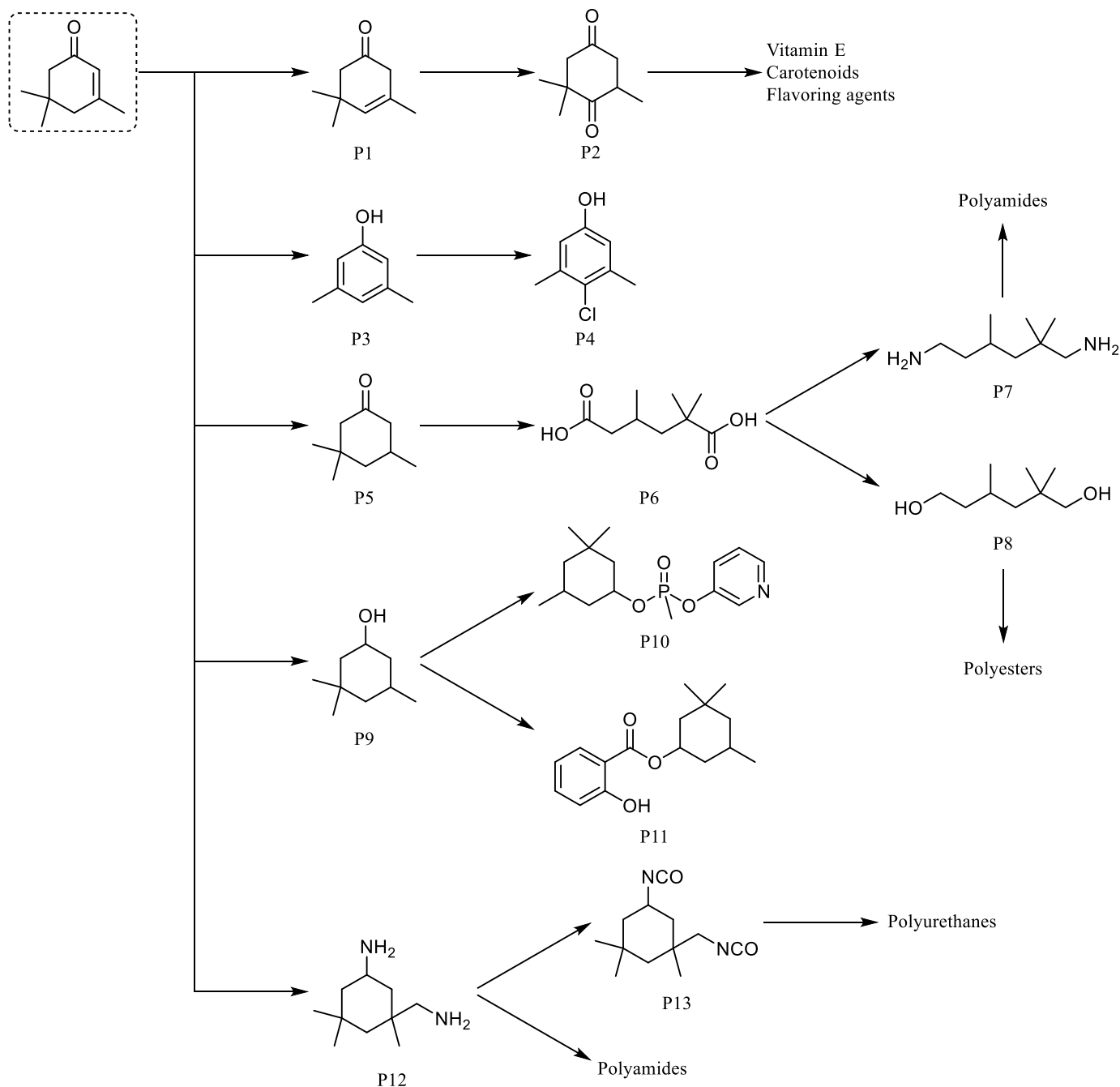


Figure 1. Products derived from IP [3,5,8,35,42,45,54,63,64,69,71,76].

Aside from these products, there are other possible applications of IP [3,32,64]: new studies, for example, report the possible use of IP as a platform chemical to produce renewable products [83], e.g., bio-based cycloalkanes as ingredients for sustainable jet fuels [84–91], often synthesized through the dimerization of IP [92].

IP is commercially obtained exclusively via self- condensation of acetone [8,64]. The starting material acetone is mainly produced via the cumene process, the world's most used process to synthesize phenol and acetone [7,93–96]. To alternatively produce renewable products from isophorone, bio-based acetone is used for the manufacturing of

isophorone [1]. The production of bio-based acetone is carried out through fermentation, e.g., the acetone-butanol-ethanol (ABE) fermentation [97–100].

While the self-condensation of acetone has been known for nearly 200 years and was first described in 1838 by Kane [101,102], the characterization of the emerging phorones, the non-cyclic trimers of acetone, and isophorone lasted until the end of the 19th century [102–105]. In 1872, for example, Kachler published the physical properties of phorones produced in different experiments [104]. The composition of the product mixture of acetone condensation depends primarily on the type of the catalyst used (basic or acidic) [5]. Kane used sulfuric acid (H_2SO_4) as an acidic catalyst to obtain a product mixture rich in mesityl oxide (MO) and mesitylene [101]. With the use of alkaline catalysts, such as potassium or sodium hydroxide, product mixtures of primarily diacetone alcohol, mesityl oxide, phorones, isophorone, and isoxylitols are formed [5,102,106–108].

The acetone condensation has a complex reaction mechanism with multiple possible reaction products, including mesityl oxide, phorone, mesitylene, isoxylitones, and ketonic resins [5,99,109–112]. In 1989, Salvapati et al. described the three-step reaction mechanism of acetone to isophorone shown in Figure 2 as the most likely reaction mechanism for the formation of isophorone [5]. Canning et al. proposed the same reaction mechanism in 2005 based on the results of experiments with deuterated acetone [113]. In addition, kinetic studies of the reaction were performed by Mei et al. [109] and others [114–122]. Mei et al. identified the formation of mesityl oxide from acetone as the rate-determining step [109], Darda et al. established the formation of diacetone alcohol from acetone [118]. In addition, Mei et al. and others found that the selectivity toward isophorone is higher than the selectivity towards higher condensation products at high temperatures [109,123].

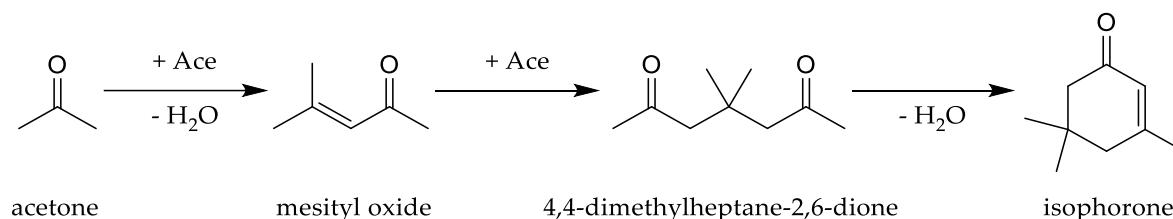


Figure 2. Reaction scheme for the synthesis of isophorone from acetone [5,113].

The three reaction steps are: 1. Aldol condensation of two molecules of acetone to mesityl oxide [5,113]; 2. Michael addition of mesityl oxide and acetone to 4,4-dimethylheptane-2,6-dione [5,113]; and 3. Intramolecular aldol condensation of 4,4-dimethylheptane-2,6-dione to isophorone [5,113].

Reaction Mechanism

Based on the published literature, the reaction network shown in Figure 3 is proposed, excluding the formation of higher condensation products such as polymers [110,124,125] or oligomers of acetone and isophorone [4,111,126–131]. The reaction starts with acetone (1), which forms diacetone alcohol (4) through aldol reaction [5,99,132,133]. From diacetone alcohol (4), mesityl oxide (3) and *iso*-mesityl oxide (2) are formed via dehydration [5,99,134–136]. The aldol reaction of diacetone alcohol (4) and acetone yields triacetone dialcohol (9) [5,99,132,137]. Triacetone dialcohol (9) mostly reverts to acetone (1) but can also form semiphorone (8) or 2,2,6,6-tetramethylpyron-4-one (14) through dehydration [137–139]. Mesityl oxide (3) and *iso*-mesityl oxide (2), the unconjugated isomer of mesityl oxide, are in equilibrium, in favor of the thermodynamically more stable mesityl oxide (3) [8,99,140]. Further reactions from acetone and mesityl oxide (3) lead to four products: 4,6-dimethylheptane-3,5-diene-2-one (5) [5,134,135], 4,4-dimethylheptane-2,6-dione (6) [5,134,135], phorone (7) [5,134,135] and semiphorone (8) [99,141]. The first three products (5–7) are assumed to be able to react further to isophorone [5,135]; however, mainly 4,4-dimethylheptane-2,6-dione (6) is expected to be the intermediate to isophorone because it rapidly converts to isophorone (11) via 1,6-aldol condensation [5,113,142]. 4,6-

Dimethylheptane-3,5-diene-2-one (5) forms isophorone (11) via an internal Michael reaction [5] and mesitylene (10) through 1,6-aldol condensation [5]. Phorone (7) reacts to isophorone (11) via 1,6 internal Michael cyclization [5] and semiphorone (8) through hydration [51,141]. Semiphorone (8) can cyclize to 2,2,6,6-tetramethylpyron-4-one (14) [5,51] and dehydrates to phorone (7) [51,99,139]. Isophorone (11) can rearrange to the constitutional isomer β -isophorone (12), and both are in equilibrium with about 97% α - (11) and 3% β -isophorone (12) [12,18,143]. α -Isophorone can react to mesitylene (10), and to 3,5-xyleneol through the loss of methane [5,144]. The isoxylitones (e.g., 15–19) are obtained through the reaction of isophorone (11) with acetone [5,51,107,109]. The isoxylitone 5,5-dimethyl-3-(2-methylprop-1-en-1-yl)cyclohex-2-en-1-one (15), for example, can also be formed via the self-condensation of mesityl oxide (3) [107,145]. 5,5-Dimethyl-3-(2-methylprop-1-en-1-yl)cyclohex-2-en-1-one (15) can further react with acetone to (E)-3-(2,4-dimethylpenta-1,3-dien-1-yl)-5,5-dimethylcyclohex-2-en-1-one (20), which is also formed through the condensation of mesityl oxide (3) with isophorone (11) [109,143]. (E)-3-(2,4-dimethylpenta-1,3-dien-1-yl)-5,5-dimethylcyclohex-2-en-1-one cyclizes to 3,3,6,8,8-pentamethyl-3,4,7,8-tetrahydronaphthalen-1(2*H*)-one (21), which forms the insecticide 3,3,6,8-tetramethyl-3,4-dihydronaphthalen-1(2*H*)-one (22) through the loss of methane [109,128,146,147].

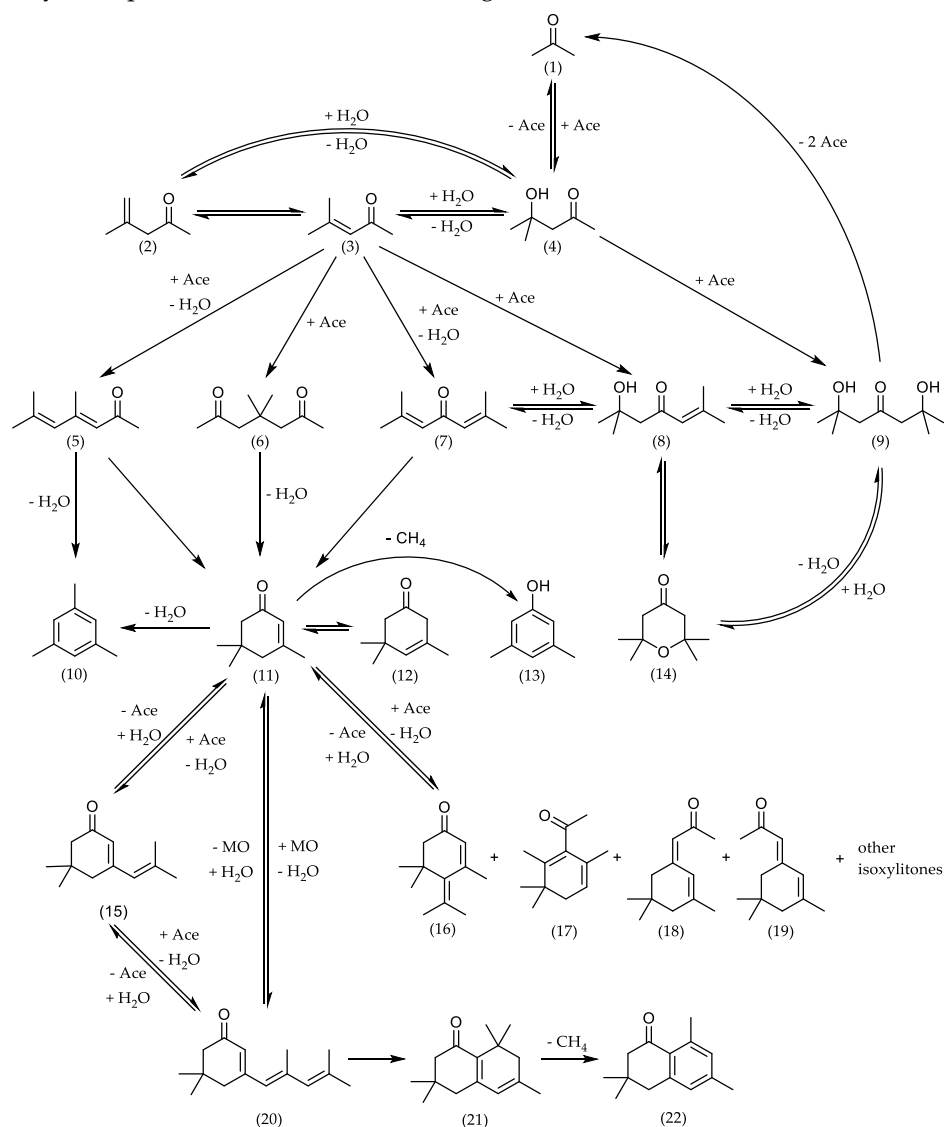


Figure 3. Reaction network of the self-condensation of acetone [4,5,99,107,109,113,123,134–139,141,143,144].

4. Industrial Manufacturing of Isophorone

Today, isophorone is produced via liquid or vapor-phase base-catalyzed self-condensation of acetone [8,64,106,111,126,127,131,148,149]. The liquid-phase process has been known at least since 1941, with aqueous KOH or NaOH being used as catalysts [150]. However, the selectivity to isophorone is often less than 70% [8] because acetone and isophorone react to higher condensation products [5,8,107,110,124,128,151]. The vapor-phase condensation over heterogeneous solid base catalysts has been studied for over 80 years [152] and is considered to be the greener process [109], since the selectivity toward isophorone can be higher than the selectivity in the liquid-phase process and since no alkaline wastewater is produced [8,109], although the energy efficiency of the vapor phase process is lower [8]. To prevent the deactivation of the catalyst through coke formation, the acetone conversion is limited to a low level [109,153]. In the early years, calcium carbide (CaC_2) was used as a catalyst, while mainly metal oxides are used today to catalyze the reaction [5,106,152].

Whereas the total production output of isophorone was 15,000 to 20,000 t/y in the mid-1960s, it had more than doubled in the year 2000 with approx. 50,000 t/y [64], and had doubled again to 100,000 t/y in 2005 [6]. In 2016, the production volume in the United States amounted to 23,000 t/y [149]. The current isophorone market value is estimated to be worth USD 1.25 billion in 2022 with a compound annual growth rate of about 5% [9,10]. Some of the leading producers of isophorone and isophorone-related products such as IPDA and IPDI are Arkema SA, BASF SE, Covestro AG, and Evonik Industries AG [6,9,10,154–160].

4.1. Liquid-Phase Process

The liquid-phase condensation, known as the Scholven process [128], is primarily catalyzed using homogenous alkaline catalysts, mainly aqueous sodium hydroxide or potassium hydroxide solutions [5,106]. For the liquid-phase condensation, the typical reaction temperature is between 205 and 250 °C [8,161] and the pressure is approx. 3.5 MPa [8]. A process scheme for the liquid-phase condensation of acetone to isophorone is depicted in Figure 4.

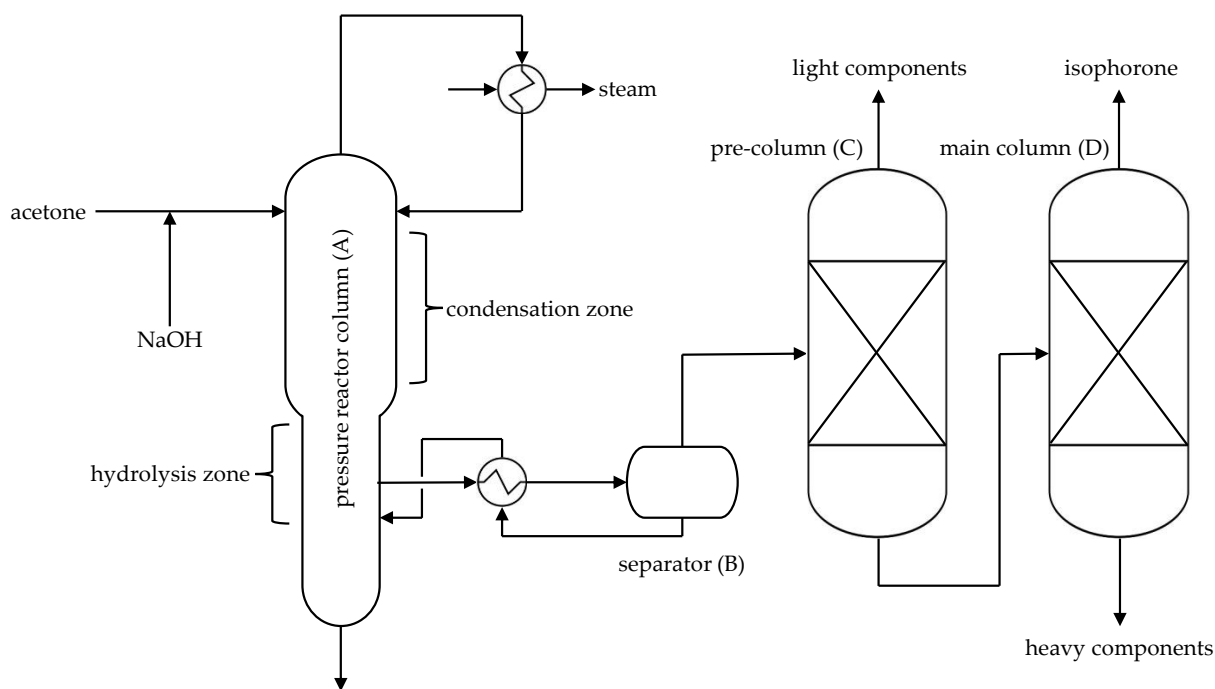


Figure 4. Process scheme of the liquid-phase production of isophorone [3,4,8,162].

The pressure column (A) with head conditions of approx. 205 °C and approx. 3.5 MPa is fed with a mixture of acetone and an aqueous alkaline solution [8,106]. The condensation of acetone takes place in the upper trays of the column [8]. Unreacted acetone and water

are distilled overhead as an azeotropic mixture, which is afterwards recycled to the column [8]. High-boiling-point compounds move down the column, where, in the lower part of the column, by-products are cleaved into isophorone and acetone via alkali-promoted hydrolysis [8]. The mixture leaving the column is separated (B) into an organic and an aqueous layer and the latter is recycled into the column [8]. The isophorone-rich organic phase is subsequently removed from impurities by means of distillation in two columns (C, D), leaving pure isophorone (approx. 99%) as the desired product [8]. The pre-column (C) operates at the same pressure as the reactor (A), while the main column (D) is operated at a reduced pressure [163].

To increase the isophorone yield, high-boiling side products (e.g., isoxylitones) can be hydrolyzed in a regeneration process to form acetone and isophorone [118,163–169]. Cook reported that by lowering the acetone feed rate, a higher yield can also be achieved [170].

The product quality of the isophorone can be increased by removing and purifying color-forming substances from the pressure reaction column (A) [8,163,168]. The discoloration is also carried out to increase the maximum storage time and can be achieved through various possible treatments [8] using phosphoric acid [171], an aromatic sulfonic acid such as *p*-toluenesulfonic acid [171,172], acid type fuller's earth [173], diazines [174], diisopropylamine [175], mono- and polyhydroxybenzene derivatives [176], a strong caustic aqueous solution [177] or an acidic ion-exchange resin [178].

4.2. Vapor-Phase Process

For the vapor-phase condensation, heterogeneous catalysts such as mixed metal oxide catalysts are used [179–185], e.g., lanthanum aluminum magnesium oxide [186,187] or calcium aluminum magnesium oxide [185,187]. Typical reaction conditions are a temperature of 250–350 °C and atmospheric pressure [5,8]. To prevent catalyst deactivation through coke formation, the vapor-phase reaction is operated with a low conversion rate of acetone, usually between 10 and 35% [8,148]. A process scheme for the vapor-phase condensation of acetone to isophorone is depicted in Figure 5.

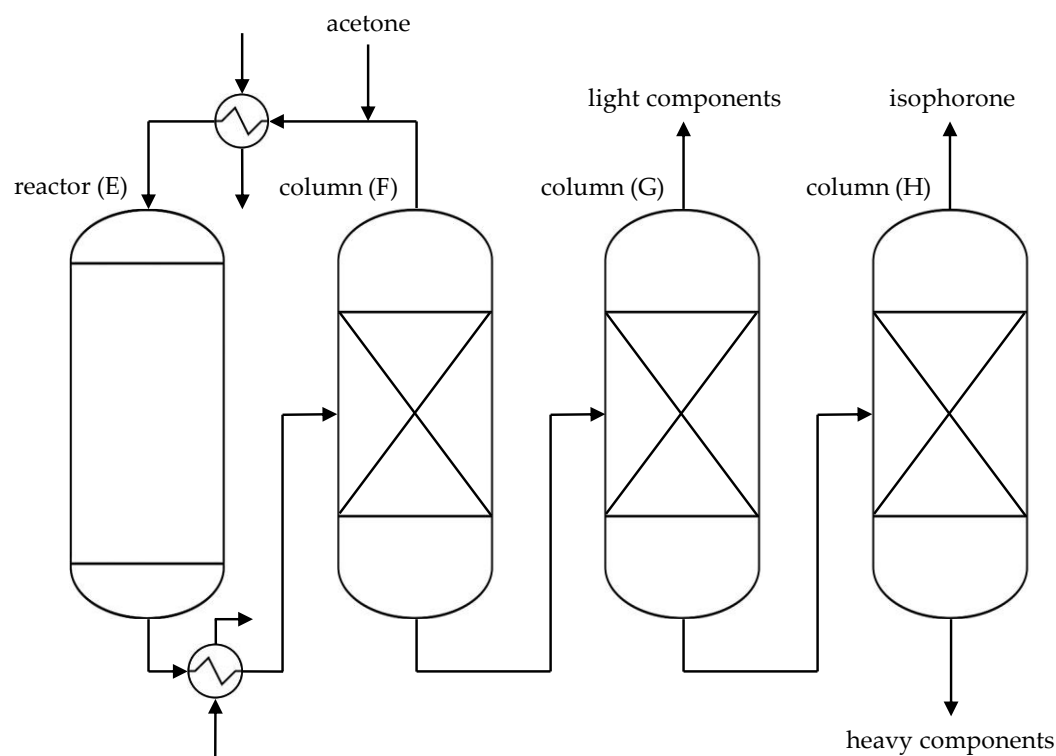


Figure 5. Process scheme of the vapor-phase production of isophorone [188].

Acetone is gasified and fed into a fixed bed reactor (E), where condensation takes place [188]. The gaseous product mixture is afterward cooled and liquefied [188]. The product mixture is pumped into a column (F), where unreacted acetone is separated from the other components [188]. The unreacted acetone is mixed with the fresh acetone, gasified, and fed back into the reactor (E) [188]. The product mixture leaving the floor outlet of column (F) is fed into another column (G) [188].

The purpose of columns G and H is identical to the purpose of columns C and D of the liquid-phase process [8,188]. In addition, the light and heavy components can partially be used for isophorone production, as in the liquid-phase process [148].

5. Catalysts

For the self- condensation of acetone to isophorone, basic catalysts are primarily used [106,148]. The acid-catalyzed condensation, with sulfuric acid or acidic zeolites, yields mesitylene as the main product [189–194]. As basic catalysts, either homogeneous or heterogeneous ones can be used [5,106,195]. In liquid-phase condensation, primarily homogeneous catalysts, such as the aqueous NaOH and KOH solutions, catalyze the reaction [8,64,148]. In the vapor-phase reaction, solid heterogeneous catalysts are used [8,64,148,196]. The uncatalyzed synthesis of isophorone is possible through electrochemical conversion [197].

5.1. Catalysts for the Liquid-Phase Condensation

Many different catalysts such as sodium ethoxide and sodium amide were used as catalysts in the early days, though the alkali hydroxides NaOH and KOH have been the primarily used catalysts in industrial isophorone production since the 1940s [102,106,112,150,198]. In the early years, the concentrations of used alkali hydroxide solutions were relatively high, at 20 to 30 wt%, and the reaction temperature was relatively low compared to later data with 150–170 °C [5,106,150,198]. Ballard et al., for example, tested concentrations of 20 to 25 wt% of NaOH solution and about 30 wt% of KOH solution at 150 to 170 °C, respectively [150,198]. They obtained a yield of up to 7% and a selectivity of up to 40% [150,198].

With higher alkali concentrations, the reaction is faster, but many by-products are formed, which is why the selectivity to isophorone is lower [5,106]. In more recent studies, therefore, the focus has been on lower alkali concentrations and higher reaction temperatures [114,161,199]. Walton and Yeomans reported a selectivity of 86% for a 0.75 wt% aqueous KOH solution at 205 °C reaction temperature [199]. Mao et al. studied the impact of different KOH concentrations (0.3–1.0 wt%), temperatures (150–250 °C), and reaction times (1–8 h) on the acetone conversion and isophorone selectivity and achieved the best result with a KOH concentration of 0.7 wt%, a reaction time of 4 h, and a temperature of 250 °C [161]. The conversion of acetone was 68% and the selectivity towards isophorone was 93% [161]. Mao et al. also tested NaOH under the same conditions [161]. While the conversion of acetone was the same (68%), the selectivity was 84%, 9% lower than for KOH [161]. Li et al. studied the impact of changing reaction conditions (catalyst dosage, temperature and pressure) for a NaOH solution used as the catalyst [114]. The best result was achieved with a reaction time of 4 h, a temperature of 220 °C, and a NaOH concentration of approx. 4 wt% [114]. The selectivity for isophorone was 81% and the yield was 53% [114]. In a continuous process with a work-up of the by-products, Chen et al. achieved a selectivity and a yield of >90% with both KOH and NaOH solutions [200]. The reactions were carried out under supercritical conditions at 320 °C and 20 MPa with 10 wt% aqueous alkali solution as catalyst [200]. The highest reported selectivity is 93% for the NaOH catalyzed reaction and 92% for the reaction catalyzed using the KOH solution [200].

He et al. used microreactor technology to optimize the synthesis of isophorone [201]. They carried out an experiment with a KOH concentration of 1.8 wt% at a temperature of 280 °C and achieved an isophorone selectivity of approx. 90% [201]. A reaction time of a few minutes as described by Chen et al. requires harsh reaction conditions of 280–320 °C and 8–20 MPa [200]. Typical reaction conditions are milder using approx. 3.5 MPa and

250 °C or even lower pressure and reaction temperature, which causes a higher reaction time between 0.5 and 4 h [8,106].

As another class of homogeneous catalysts, organic quaternary ammonium hydroxides based on imidazole (a), pyridine (b), and methenamine (c) (Figure 6) are used [202–204]. Zhou et al. tested eight of these catalysts at a temperature ranging from 150 to 180 °C with a reaction time from 5 to 8 h [202–204]. The achieved selectivity for isophorone ranged from 63 to 71%, and the conversion from 55% to 68% [202–204]. The most efficient of the tested catalysts was 1,3-di-*n*-butylimidazole hydroxide [202–204] at a reaction temperature of 160 °C and a reaction time of 7 h [202]. The reported selectivity was 71% and the conversion was 62% [202]. The advantage of these catalysts is a lower reaction temperature compared to NaOH or KOH; however, a reaction time of 5 h and more is significantly higher [202–204].

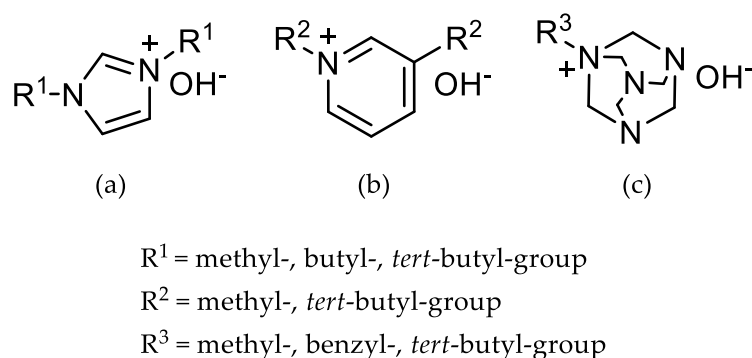
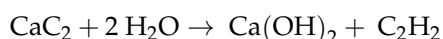


Figure 6. Organic bases used as catalysts: (a) imidazole, (b) pyridine, and (c) methenamine derivatives [202–204].

Although heterogeneous catalysts are primarily used for the vapor-phase reaction, some of these catalysts were also tested in the liquid-phase [205–208]. In the last ten years, there has been continued interest in the use of calcium carbide (CaC₂) as the catalyst in the liquid-phase condensation because it is inexpensive and acetylene is formed as a co-product [144,205,209,210]. Calcium carbide is a Lewis base catalyst [205], which was used as a catalyst for the vapor-phase condensation in the 1930s [152]. Li et al. obtained an isophorone yield of 21% and a conversion of 81% at 150 °C with CaC₂ [205].



Other heterogeneous catalysts used are metal oxides [206–208]. In the first step of a two-step process for the synthesis of isophorone, a basic anion exchange resin is used as the catalyst [206]. In the second step, a potassium hydroxide- and magnesium nitrate-doped aluminium oxide catalyst is used at 210 °C [206]. The reaction time is approx. 8 h, the achieved selectivity is up to 89%, and the reported yield is up to 71% [206]. Teissier and Kervennal used a magnesium–aluminium mixed metal oxide as the catalyst [208]. With a reaction time of 1 h and a temperature of 200 °C, they obtained a selectivity for isophorone of 51% and an acetone conversion of 31% [208]. Qiao et al. used a mixed metal oxide containing strontium, magnesium, aluminium, and praseodymium as the catalyst [207]. The continuous reactor was operated with a residence time of 1 min under supercritical conditions of 300 °C and 8 MPa [207]. Qiao et al. achieved a selectivity towards isophorone of 91% and a conversion of 41% [207].

The conversion, selectivity, and yield for the catalysts mentioned in Section 5.1 are summarized in Table 2.

Table 2. Literature acetone conversions (X), selectivity for isophorone (S) and yield (Y) for different catalysts for the liquid-phase/supercritical condensation of acetone.

Catalyst ^a	T (°C)	Reactor	Time ^b	X (%)	S (%)	Y (%)	Refs.
0.7% NaOH	250	batch	4 h	68	84	n/a	[161]
≈4% NaOH	220	batch	4 h	n/a	81	53	[114]
10% NaOH	320	continuous	2 min	n/a	93	93	[200]
20% NaOH	150	batch	3 h	n/a	39	7	[150]
25% NaOH	170	continuous	37 min	n/a	n/a	4 ^c	[198]
0.7% KOH	250	batch	4 h	68	93	n/a	[161]
0.75% KOH	≈205	continuous	n/a	n/a	86	7	[199]
1.8% KOH	280	continuous	5 min	>20	>90	n/a	[201]
10% KOH	300	continuous	1 min	n/a	92	91	[200]
30.3% KOH	169	continuous	17.5 min	n/a	n/a	6	[198]
Methylmetheneamine hydroxide	180	batch	5 h	55	63	n/a	[203]
Benzylmetheneamine hydroxide	150	batch	6 h	59	65	n/a	[203]
<i>tert</i> -Butylmetheneamine hydroxide	160	batch	7 h	60	66	n/a	[203]
1,3-Dimethylimidazole hydroxide	180	batch	5 h	62	71	n/a	[202]
1,3-Di- <i>tert</i> -butylimidazole hydroxide	150	batch	6 h	67	69	n/a	[202]
1,3-Di- <i>n</i> -butylimidazole hydroxide	160	batch	7 h	68	71	n/a	[202]
N,3 Dimethylpyridine hydroxide	150	batch	6 h	55	63	n/a	[204]
N- <i>tert</i> -butyl-3-methylpyridine hydroxide	160	batch	7 h	57	62	n/a	[204]
CaC ₂	150	batch	2 h	81	n/a	21	[205]
Al ₂ O ₃ , basic resin	210	batch	7–8 h	n/a	89	71	[206]
Mg _{1-x} Al _x O _{1+x}	200	batch	1 h	38	51	n/a	[208]
Sr _{0.1} Mg _{0.6} Al _{0.15} Pr _{0.05} O	270	continuous	1 min	41	91	n/a	[207]

^a Percentage refers to weight percentage (wt%). ^b Residence time for continuous and reaction time for batch.
^c Mass fraction of isophorone.

Although corrosion can be avoided using heterogeneous catalysts, isophorone continues to be industrially produced using aqueous alkali hydroxide solutions in the liquid-phase [106]. The main reason for this is that this process has been continuously optimized and is currently very efficient [11,148,163–167,171,199–201,211–216]. For the switch to the vapor-phase process to be profitable, it must be more efficient or less expensive than the liquid-phase process. In addition, the service life of the heterogeneous catalysts must be high. Over the years, however, various companies have also been established new patents on vapor-phase processes with improved processes [152,217–227].

5.2. Catalysts for the Vapor-Phase Condensation

While in the 1930s, calcium carbide, calcium oxide, and calcium hydroxide were used as catalysts for the vapor-phase condensation, mostly mixed/composite metal oxides, often containing aluminum and magnesium, are used as catalysts nowadays [5,106,152]. While pure magnesia can be used as a catalyst, the reported selectivity toward isophorone is 35 % and below [5,228]. A significant increase in selectivity is possible through the combination of magnesium with other metals, mostly aluminum [106].

Due to the high catalytic performance of magnesium–aluminum mixed oxide and its low cost, this is probably the most common catalyst for the vapor-phase reaction [106]. Magnesium–aluminum mixed oxide is often synthesized using the coprecipitation method via calcination of hydrotalcite [229–232]. The investigations with magnesium–aluminum

mixed oxide focus on the temperature range from 250 to 300 °C, with a residence time of 30 to 60 min [184,186,229–233]. A selectivity range of approx. 20 to 95% is reported [184,186,229–233]. Ordóñez et al. tested a temperature range of 50 to 450 °C and achieved the best selectivity to isophorone of 23% at 250 °C [229]. Kelkar and Schutz reported their highest selectivity toward isophorone to be 75% at 250 °C, 25% acetone conversion, and a catalyst particle size of 0.315 mm [230]. They investigated the temperature range from approx. 250 to 315 °C and particle sizes from 0.315 to 6.35 mm [230]. Gao reached a selectivity of 65% at 250 °C with Mg–Al mixed oxide as the catalyst [231]. The regenerated catalyst compared to fresh catalyst showed a selectivity decrease of approx. 5% [231]. Other data for the Mg–Al mixed oxide as the catalyst was published by Ma et al., Li et al., and Liu et al. [184,232,233]. Ma et al. reported a selectivity of 86%, a yield of 73%, and a conversion of higher than 70% at 250 °C [232], Li et al. a selectivity of 95% and a conversion of 36% at 290 °C [184], and Liu et al. a conversion of 20% and a selectivity of 56% at 300 °C [233].

Other catalysts mentioned in the literature are mixed oxides of magnesium or magnesium and aluminum with other metals [181,185–187,227,234–236]. Zhang et al. prepared magnesium–aluminum–yttrium-layered double-oxides with different Y-doping [234]. With a doping quantity of 1% they achieved a selectivity of 59% and a yield of 22% at 300 °C [234]. Other metals that were used for mixed metal oxides are chromium, zirconium, lanthanum, calcium, barium, and cerium in different combinations [185–188,227,235]. The reaction temperature for the different experiments were usually between 250 and 450 °C [185–188,227,235]. The reported selectivity is about 60 to 80% [185–188,227,235]. An especially high selectivity was reported by Zhao et al. They prepared a Ca–Zr–Mg–Al mixed oxide catalyst, which achieved a selectivity of up to 97% at 300 °C and 1 MPa with an acetone conversion of 34% [227]. These results are part of a stability test to demonstrate the stability of the catalyst over 1000 h [227]. After the 1000 h, the selectivity was still over 90%. The highest selectivity of 97% was measured after about 600 h [227].

In addition, data on several other catalysts have also been published, including molecular sieves [237], cesium on carbon [238], CsOH/SiO₂ [113], Na⁺/TiO₂ [239], Na⁺-(VO)₂P₂O₇ [240], NaOH [241], Ca(OH)₂–CaO [242] and Ca(OH)₂ [152]. Ma et al. used a modified X molecular sieve as a catalyst and achieved a selectivity of 52% [237]. Stevens et al. prepared cesium on nanoporous carbon as the catalyst [238]. The reported selectivity is 61% [238]. Canning et al. used cesium hydroxide on silica for their mechanistic study and achieved a selectivity of 19% [113]. Zamora et al. tested sodium–chloride-doped titania as the catalyst [239]. The selectivity was 4% [239]. Thomas et al. studied the aldol condensation of acetone with different alkali-modified vanadium phosphates as catalysts [240]. With the most efficient catalyst, a sodium-modified vanadium phosphate, they achieved a selectivity for isophorone of 95% [240]. Qian et al. used molten NaOH as the catalyst [241]. The published selectivity is 60% [241]. Wang et al. prepared a catalyst containing Ca(OH)₂ and CaO through the calcination of Ca(OH)₂ [242]. The catalyst, prepared with a calcination temperature of 550 °C, achieved a selectivity higher than 80% [242]. For pure Ca(OH)₂ as the catalyst, Vaughn et al. reported as yield of 25% [152].

The conversion, selectivity, and yield for the catalysts mentioned in Section 5.2 are summarized in Table 3.

Table 3. Literature acetone conversions (X), selectivity for isophorone (S) and yield (Y) for different catalysts for the vapor-phase condensation of acetone.

Catalyst	T (°C)	Reactor	Residence Time	X (%)	S (%)	Y (%)	Ref.
Magnesium oxide	450	continuous	n/a	37	33	n/a	[228]
Mg–Al mixed oxide	250	continuous	n/a	7	23	n/a	[229]
Mg–Al mixed oxide	250	continuous	30 min	25	75	n/a	[230]
Mg–Al mixed oxide	250	continuous	1 h	20	65	n/a	[231]

Table 3. Cont.

Catalyst	T (°C)	Reactor	Residence Time	X (%)	S (%)	Y (%)	Ref.
Mg–Al mixed oxide	250	continuous	1 h	n/a	86	73	[232]
Mg–Al mixed oxide	290	continuous	n/a	36	95	n/a	[184]
Mg–Al mixed oxide	300	continuous	n/a	20	56	n/a	[233]
Mg–Al–Y-layered double-oxide	300	continuous	9.5 min	38	59	22	[234]
Cr–Mg–Al mixed oxide	240	continuous	n/a	25	74	n/a	[181]
Zr–Mg–Al mixed oxide	240	continuous	n/a	37	73	n/a	[181]
La–Mg–Al mixed oxide	250	continuous	1 h	29	64	19	[186]
La–Mg–Al mixed oxide	250	continuous	37.5 min	41	72	29	[187]
Ca–Mg–Al mixed oxide	250	continuous	37.5 min	28	80	23	[187]
Ca–Mg–Al mixed oxide	250	continuous	1 h	55	65	36	[185]
Ca–Zr–Mg–Al mixed oxide	300	continuous	30 min	35	97	n/a	[227]
Mg–Zr mixed oxide	450	continuous	n/a	43	9	n/a	[235]
Mg–Al–Ca–Ba–Zr–Ce mixed oxide	300	continuous	n/a	86	n/a	76	[188]
molecular sieve	350	continuous	30 min	26	52	n/a	[237]
Cs/nanoporous carbon	225	continuous	n/a	12	61	n/a	[238]
CsOH/SiO ₂	400	continuous	n/a	3	19	n/a	[113]
Na ⁺ /TiO ₂	300	continuous	n/a	20	4	n/a	[239]
Na ⁺ -(VO) ₂ P ₂ O ₇	400	continuous	n/a	7	>95	n/a	[240]
NaOH	220–350	continuous	n/a	32	60	n/a	[241]
Ca(OH) ₂ –CaO	550	continuous	n/a	47	>80	n/a	[242]
Ca(OH) ₂	350	continuous	n/a	n/a	n/a	25	[152]

In addition to the 47 literature results presented in this review, many other studies have been conducted [243–259] and many other patents have been published [260–269].

6. Conclusions

The self-condensation of acetone has been known for nearly 200 years and with a growing production volume of acetone, it represents a suitable way to upgrade acetone. Isophorone is one of the main products of this reaction and is already used industrially for various purposes. Due to the complex reaction mechanism, very different product mixtures with widely varying isophorone yields are formed under different reaction conditions and with different catalysts. Therefore, the key to optimize the isophorone selectivity of this process is the optimization of the reaction parameters.

Today, the liquid-phase process is the one most commonly operated on a production scale; however, the disadvantage of the liquid-phase process is corrosion, while the vapor-phase process has a higher energy demand. Switching from the liquid-phase to the vapor-phase process is therefore not yet profitable. However, a selectivity of more than 90% for isophorone was reported for both vapor-phase and supercritical condensation.

The trend over the last few years of improving the selectivity toward isophorone seems to develop toward short reaction times and high temperatures. This can also be observed for the heterogeneous catalyst Sr_{0.1}Mg_{0.6}Al_{0.15}Pr_{0.05}O, which showed a selectivity of 91.2% at 1 min reaction time under supercritical conditions.

In the vapor-phase, catalysts, e.g., Mg–Al mixed oxide and Ca–Zr–Mg–Al mixed oxide achieved a selectivity toward isophorone of more than 90%, too. Additionally, long-term tests over 1000h were carried out with these catalysts, which showed that there was no catalyst deactivation due to coking.

However, whether these vapor-phase processes will become more attractive in the future ultimately depends on whether these processes can be operated more cheaply and/or more easily than the established liquid-phase processes. Future developments will probably focus on further improving selectivity and on improving process efficiencies, e.g., by increasing the acetone conversion while maintaining a high selectivity.

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