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# Selective removal of chlorinated impurities from (R)-Carvone using an industrial alkaline sulfite process

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**Abstract**: Chlorinated impurities present in crude (*R*)-Carvone, ($C_{10}H_{14}O$), preferred IUPAC name 2-Methyl-5-(prop-1-en-2-yl) cyclohex-2-en-1-one, resulted in chemical synthesis, represent a significant challenge for its utilization, as they may compromise product stability, safety and applicability in food, fragrance and pharmaceutical formulations. This study presents an industrial validated process, for the selective removal of chlorinated compounds, using an alkaline sulfite system, under moderate reaction conditions. The method employs a biphasic mixture of sodium hydroxide (NaOH), sodium sulfite ($Na_2SO_3$), isopropanol ($C_3H_8O$) and controlled acetic acid ($C_2H_4O_2$) addition, in a stirred reactor, equipped with reflux and solvent recovery units, followed by solvent distillation and recycling steps. Process operates at temperatures between 60 and 90$^0$C and includes a closed loop recycling stage, which improves reagent utilization and reduces waste generation. The proposed approached enables efficient conversion and removal of chlorinated terpene impurities, while preserving the structural integrity of the targeted ketone. The integration of reaction and purification in a single industrial unit, combined with solvent recovery and neutralization stages, provides a scalable and economically attractive solution for the production of hi purity (*R*)-Carvone. This work demonstrates a practical and selective dichlorination strategy suitable for large scale terpene processing.

**Keywords**: carvone, dichlorination, impurities, ketones, limonene, terpene.

## • Introduction

Carvone is an optically active monoterpene ketone widely used in food, fragrance, cosmetic and pharmaceutical industries due to its characteristic aroma and biological activity. It is naturally present in spearmint oil, while industrial production relies on the transformation of renewable terpene feedstocks such as limonene derived from citrus byproducts.

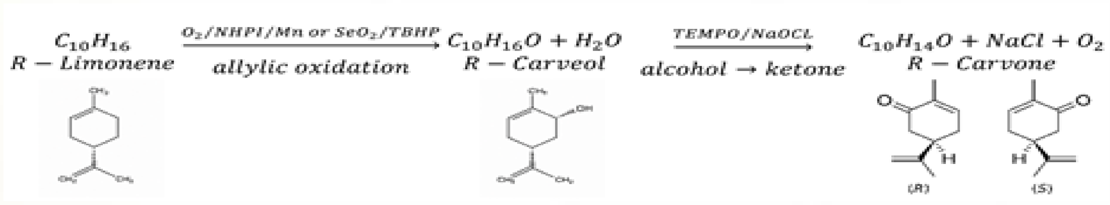


*Fig.1 Conversion of R-Limonene to (R)-Carvone*

These processes may generate chlorinated terpene impurities, which can negatively affect product stability, odor and regulatory compliance. This work presents an alkaline sulfite-based treatment for selective removal of chlorinated impurities, enabling efficient large scale production of high-purity (R)-carvone.

## • Results and discussions

The process demonstrated high reproducibility, with minimal material losses (0.05-0.2% per batch) and consistent (*R*)-carvone purity of 98.0-99.2%, confirming stable industrial performance. Chlorine content and product quality were validated using micro coulometry (AQF-2100H), ion chromatography (ICS-3000), and GC (Agilent 7890A), ensuring reliable analytical control.

**Table 1.** Table 1. Elimination Cl form (*R*)-Carvone

| Batch No | Crude (R)-carvone (kg) | Initial Cl (ppm) | Final Cl (ppm) | Cl removal (%) | Final purity (%) | (R)-carvone Final (kg) |
|---|---|---|---|---|---|---|
| 1 | 800 | 1278 | 23 | 98,2 | 98.6 | 799.5 |
| 2 | 800 | 1342 | 32 | 97,6 | 98.8 | 798.9 |
| 3 | 800 | 1450 | 31 | 97,9 | 98 | 799 |
| 4 | 800 | 1258 | 29 | 97,7 | 98.1 | 797.9 |
| 5 | 800 | 1354 | 30 | 97,8 | 98.2 | 798.6 |
| 6 | 800 | 1284 | 28 | 97,8 | 98.4 | 799.6 |
| 7 | 800 | 1489 | 36 | 97,6 | 98.4 | 798.7 |
| 8 | 800 | 1254 | 24 | 98,1 | 98.1 | 795.6 |
| 9 | 800 | 1365 | 32 | 97,7 | 99.2 | 799.4 |
| 10 | 800 | 1701 | 24 | 98,6 | 98.6 | 798.6 |
| 11 | 800 | 1389 | 28 | 98,0 | 98.6 | 799.2 |
| 12 | 800 | 1462 | 31 | 97,9 | 98.4 | 798.1 |
| TOTAL | 9600 | | | | | 9583.1 |

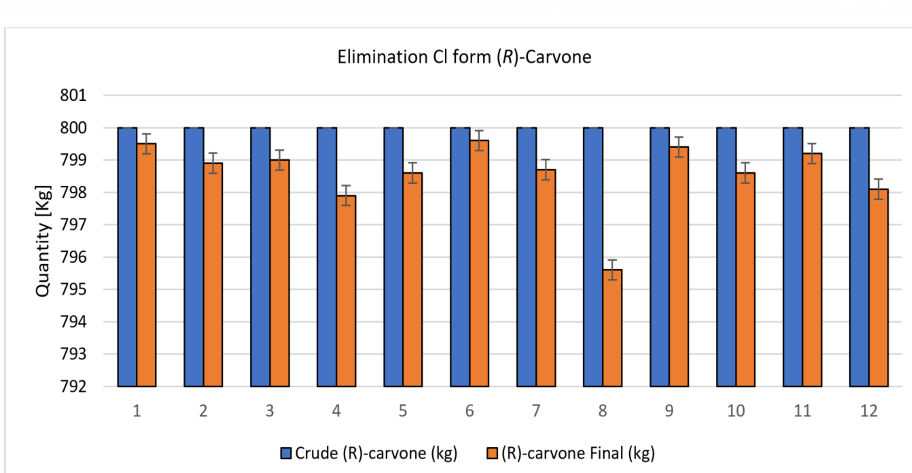


**Figure 3.** Elimination Cl form (*R*)-Carvone final results

Furthermore, the relatively narrow variation in chlorine removal efficiency across all batches indicates a high degree of process reproducibility under the applied operating conditions. This consistency confirms the robustness of the developed methodology, supporting its suitability for scale-up and reliable industrial implementation.

## • Material and metod

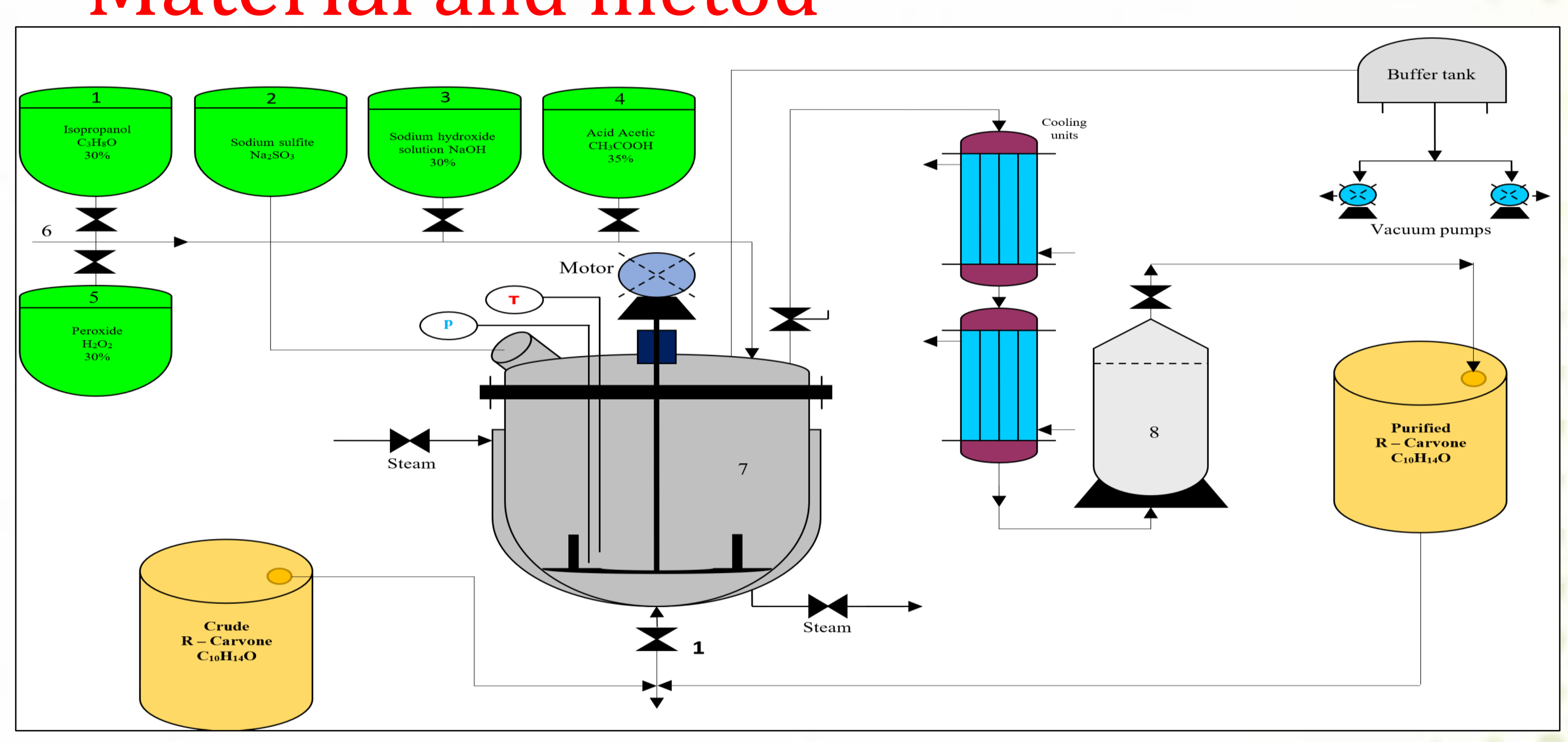


Fig 2. Industrial continuous installation for eliminating Cl form (R)-Carvone

Crude (R)-carvone is treated in a stirred reactor with $Na_2SO_3$/NaOH in isopropanol, followed by controlled acid addition and heating to remove chlorinated impurities. The mixture undergoes distillation and phase separation, with an additional recycling stage (reflux and fractional distillation) to improve purity and yield. Final neutralization with $H_2O_2$ and acetic acid ensures pH stabilization and production of high-purity (R)-carvone.

## • Conclusions

- The alkaline sulfite system establishes a reactive equilibrium between sulfite and bisulfite species, enabling efficient interaction with chlorine containing impurities;
- Reactive chlorine species are reduced to stable chloride ions, while organochlorine compounds are converted into more polar and less reactive derivatives;
- Process conditions, including controlled acidification, temperature, and mixing, enhance reaction kinetics and promote chemical stabilization prior to separation;
- Phase separation and distillation effectively remove aqueous phase impurities, allowing recovery of an organic phase enriched in high purity (*R*)-carvone;
- Final oxidative neutralization ensures complete stabilization of residual species, resulting in a robust, scalable, and efficient dichlorination process.

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