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## Systematic Review of GC-MS-Based Methods for Determination of Aldehydes in Electronic Cigarette Liquids

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**Abstract:** Electronic cigarettes generate toxic aldehyde compounds through the thermal decomposition of primary *e-liquid* constituents, namely propylene glycol and glycerol, yet standardized analytical methodologies for their determination remain critically fragmented across research institutions worldwide. This study conducted a systematic literature review adhering to the PRISMA 2020 protocol, encompassing 15 peer-reviewed articles retrieved from PubMed, Scopus, and Web of Science covering the period 2016 to 2024, specifically examining GC-MS-based methods for aldehyde determination in *e-liquid* matrices. Comparative synthesis demonstrated that headspace GC-MS incorporating isotopic internal standards constitutes the most robust analytical approach, consistently achieving linearity coefficients exceeding  $R^2 > 0.990$  without the requirement for chemical derivatization. Formaldehyde, acetaldehyde, and acrolein were recurrently identified as predominant aldehydes, with emission intensities exhibiting strong positive correlation with device operational parameters including voltage and heating power. Detection of formaldehyde-hemiacetal revealed substantial underestimation inherent to conventional measurement approaches regarding total aldehyde exposure. Pronounced methodological fragmentation across laboratories and the absence of matrix-specific certified reference materials represent principal barriers to global interlaboratory data harmonization. Standardization of reference methods through international interlaboratory collaboration is critically recommended to ensure robust and effective safety surveillance of electronic cigarette products.

**Keyword:** Gas Chromatography-Mass Spectrometry, aldehydes, electronic cigarette e-liquid

### INTRODUCTION

The use of electronic cigarettes has experienced a rapid surge worldwide in the past decade, including in Indonesia, as public perception has grown that these products are a safer alternative to conventional cigarettes. However, this assumption is now increasingly being

scientifically questioned. Electronic cigarette liquids, known as e-liquids, contain a primary mixture of propylene glycol (PG) and glycerol (VG), which when heated undergo thermal decomposition and produce various hazardous compounds, including aldehydes. Aldehydic compounds such as formaldehyde, acetaldehyde, and acrolein have been identified as toxic and carcinogenic agents with the potential to seriously damage the respiratory tract and other vital organs. Both main components of e-liquids, PG and glycerol, have been shown to produce formaldehyde and acetaldehyde as thermal decomposition products that are highly hazardous to human health (Ferney et al., 2025).

Various analytical techniques have been developed to detect and quantify aldehyde compounds in e-liquid matrices and e-cigarette aerosols. Commonly used methods include High-Performance Liquid Chromatography (HPLC) with derivatization using the reagent 2,4-dinitrophenylhydrazine (DNPH) and ultraviolet spectrophotometry. However, these methods have several fundamental weaknesses, including limited selectivity due to interference from flavor compounds in e-liquids and the inability to simultaneously identify volatile compounds in complex matrices. This is where Gas Chromatography-Mass Spectrometry (GC-MS) emerges as a far superior analytical solution, capable of simultaneously separating, identifying, and quantifying volatile compounds with high levels of sensitivity and selectivity. GC-MS has even been recommended as the most appropriate instrument for the analysis of volatile organic compounds in the context of e-cigarettes, with its ability to characterize hundreds of components simultaneously in a single analysis (Ferney et al., 2025).

However, significant gaps exist in the currently available scientific literature. Existing studies on methods for analyzing carbonyls, including aldehydes, in e-cigarette aerosols are fragmented and methodologically inconsistent. Most studies evaluate only one sample collection method without comparing the performance of different approaches, making it difficult for researchers and regulators to determine the most reliable method standards. Furthermore, in-depth evaluation of experimental quality and validation parameters such as limit of detection (LOD), limit of quantification (LOQ), linearity, and precision are rarely systematically conducted. This situation is exacerbated by the fact that the few available studies demonstrate a lack of adequate experimental reproducibility, questioning the validity of the resulting data (Sussman et al., 2024). Furthermore, flavor compounds in e-liquids have been shown to create significant analytical interferences with HPLC-UV methods, but how GC-MS specifically addresses this challenge in e-liquid matrices has not been comprehensively and systematically studied (Noël & Ghosh, 2022).

The novelty of this study lies in its systematic review approach of GC-MS-based methods specifically for the determination of aldehydes in e-cigarette e-liquids, an approach that has not been previously attempted in the available scientific literature. All relevant studies were collected, screened, and methodically analyzed based on strict inclusion and exclusion criteria, allowing for the identification of current analytical trends while simultaneously identifying knowledge gaps that need further investigation. This approach differs from existing narrative reviews by providing a structured evaluation of the analytical performance of various GC-MS method variants applied specifically to e-liquid matrices (Carbonyls & Methods, 2023).

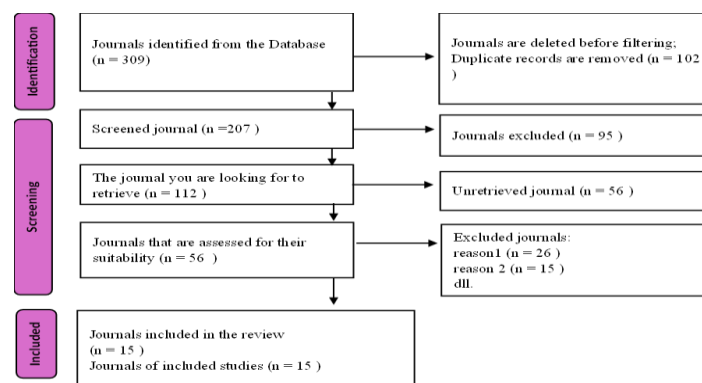
Based on the description above, the problem formulation in this study is how are the characteristics, analytical performance, and validity of various GC-MS-based methods that have been developed for the determination of aldehydes in e-cigarette e-liquids based on a systematic review of available scientific literature? The purpose of this study is to systematically identify, analyze, and synthesize various GC-MS approaches in determining aldehyde levels in e-liquids, in order to produce comprehensive methodological recommendations for researchers, regulators, and industry in supporting more effective and standardized safety supervision of e-cigarette products.

## METHOD

This study employed a Systematic Literature Review (SLR) approach designed to collect, filter, and synthesize scientific evidence in a structured and transparent manner. The reporting process for this systematic review adheres to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) 2020 guidelines, which are cutting-edge reporting standards widely adopted in systematic review-based research across various disciplines, including analytical science and tobacco toxicology. The PRISMA 2020 guidelines include a 27-point checklist that guides transparent reporting of each review stage, from data source identification to final results synthesis, enabling the research process to be replicated and verified by other researchers (Page et al., 2021). A systematic literature search was conducted through three reputable electronic databases: PubMed, Scopus, and Web of Science, with a publication period limited from 2016 to 2024 to ensure the relevance and currency of the data obtained. The keywords used included combinations of terms such as "GC-MS," "gas chromatography-mass spectrometry," "aldehydes," "formaldehyde," "acetaldehyde," "acrolein," "e-liquid," "electronic cigarette," and "vaping," which were linked using the Boolean operators AND and OR to optimize the search results. The entire search process resulted in a total of 309 articles identified from these various databases.

The article selection process involved two main phases, illustrated in the PRISMA flowchart in Figure 1. In the first phase (identification), 102 duplicate articles were removed, leaving 207 articles for further screening. In the second phase (screening), the titles and abstracts of these 207 articles were independently evaluated, and 95 articles were excluded due to lack of topical relevance. Of the 112 articles that passed the initial screening, full-text retrieval was performed, but 56 articles were inaccessible or unavailable in full, leaving 56 articles for in-depth eligibility assessment. From this eligibility assessment, 41 articles were further excluded based on predetermined reasons, including 26 articles for not specifically using the GC-MS method (Reason 1) and 15 articles for not focusing on the determination of aldehydes in e-liquids (Reason 2), among several other articles. Ultimately, 15 articles were determined to meet all inclusion criteria and were included in the synthesis of this review.

The inclusion criteria applied included: articles published in peer-reviewed scientific journals, using GC-MS as the primary analytical method, specifically targeting aldehyde compounds as analytes in e-liquid matrices, and being available in English with accessible full-text. Conversely, exclusion criteria included review articles, conference proceedings, studies that only analyzed aerosols without directly involving e-liquids, and studies that did not adequately report method validation parameters. Data extraction from the 15 selected articles was performed in a standardized manner using a form containing information on the author's name, year of publication, type of GC-MS instrument, sample preparation technique, target aldehyde compound, analytical conditions, and reported validation parameters. All extracted data were then synthesized narratively and comparatively to identify methodological patterns, strengths, and limitations of each approach studied.



Picture 1. Flowchart Prisma

## RESULTS AND DISCUSSION

Based on a systematic selection process following the PRISMA process as described in the methods section, 15 scientific articles were obtained that met all inclusion criteria and were subsequently used as primary sources in this review. All articles were published in internationally reputable scientific journals covering the period 2016 to 2024. The selected articles discuss various analytical aspects related to the detection of hazardous volatile compounds—including aldehydes—in e-liquids and e-cigarette aerosols, using various methods based on gas chromatography combined with mass spectrometry (GC-MS). The diversity of analytical approaches, sample matrices, target compounds, and instrumentation conditions used in these 15 studies provides a comprehensive overview of the evolving methodological landscape in this field. A summary of the characteristics of all included articles is presented in Table 1 below.

**Table 1. Synthesis of Study Results from 15 Reviewed Journals**

No	Author & Year	Title (Abbreviated)	Analytical Methods	Target Compound	Matriks	Key Findings
1	(Baldvinos et al., 2024)	Vape condensate collection for degradant identification	GC-MS + toxicity test	Volatile degradation compounds	Aerosol/ <i>condensate</i>	Alternative aerosol capture methods are able to identify products degradasi in vitro lung model
2	(Sleiman, 2016)	Emissions from Electronic Cigarettes: Key Parameters	GC-MS	<i>Formaldehyde, acetaldehyde, acrolein, glycidol</i>	Aerosol/ <i>e-liquid</i>	Aldehyde emissions increased 3× when the voltage increased from 3.3 to 4.8 V; acrolein increased 10×
3	(Eshraghi an & Al-delaimy, 2021)	Review of constituents in e-cigarette liquids and aerosols	Review multi-metode	60 compounds ( <i>e-liquid</i> ), 47 ( <i>aerosol</i> )	<i>E-liquid &amp; aerosol</i>	Teridentifikasi 22 senyawa bersama termasuk <i>acetaldehyde</i> dan <i>formaldehyde</i>
4	(Dagla, 2023)	Two Fast GC-MS Methods for E-Cigarette Refill Liquids	GC-MS (2 <i>method</i> )	Nikotin, PG, VG, <i>diacetyl, ethylmaltol</i>	<i>E-liquid</i>	Two fully validated GC-MS methods; there are differences in levels between label and measured results.
5	(Golpe et al., 2023)	Volatile & semi-volatile compounds in e-liquids by GC-AccMS	GC-TOF-MS ( <i>non-target screening</i> )	>250 compounds including aldehydes & acetals	<i>E-liquid</i>	Identified >250 compounds; PG acetals with flavor aldehydes reach a ratio of 2–80%
6	(Krüseman et al., 2020)	GC-MS C of e-cigarette refill solutions	GC-MS	79 senyawa <i>flavoring</i>	<i>E-liquid</i>	Average 6±4 <i>flavoring</i> compounds/product ; validation includes linearity, sensitivity, accuracy
7	(Lebouf, 2018)	Headspace analysis of VOCs in e-liquid bulk	<i>Static headspace</i> GC-MS	20 VOC + senyawa <i>tentatif</i>	<i>E-liquid</i> (146 sampel)	Sensitive static headspace method without derivatization; benzene detected at

No	Author & Year	Title (Abbreviated)	Analytical Methods	Target Compound	Matriks	Key Findings
						alarming concentrations
8	(Ogunwale et al., 2017)	Aldehyde Detection in Electronic Cigarette Aerosols	GC-MS + <i>silicon microreactor</i> (AMAH)	<i>Acetaldehyde, acrolein, formaldehyde</i>	Aerosol	New generation devices produce higher aldehydes; formaldehyde-hemiacetal detected at $\geq 11.7$ W
9	(Sala et al., 2017)	Dynamic measurement of carbonyl compounds in e-cig vapors	GC-MS + SPME <i>on-fiber derivatization</i>	Senyawa karbonil/aldehida	Aerosol/vapor	New carbonyl compounds are formed during vaping; formation is dependent on device and puff topography.
10	(Telgheder et al., 2023)	Simple & sensitive e-cigarette aerosol sampling for GCxIMS & GC-MS	GC-MS + GCxIMS ( <i>headspace vial dinging</i> )	26 flavor compounds + degradation products	Aerosol	The HS-GC-MS method is simple and inexpensive; it allows the identification of unknown compounds.
11	(Wang et al., 2024)	TSNA in e-cigarette liquids by UPLC-QTOF-HRMS	UPLC-QTOF-HRMS	4 TSNA (NNN, NNK, NAT, NAB)	<i>E-liquid &amp; aerosol</i>	Very low LOQ (0.001–0.017 ng/g); the main source of TSNAs is tobacco extract.
12	(Wei et al., 2021)	Volatile compounds in cigarette adhesive by HS-GC-MS	HS-GC-MS	11 volatile compounds including aldehydes	Cigarette adhesive	Linearity $R^2 > 0.9932$ ; LOD 3.1–147.7 ng/g; RSD < 19.8%; recovery 68.1–108.3%
13	(Yang et al., 2023)	Pyrolysis products of nicotine salts by Py-GC/MS	<i>Pyrolysis-GC/MS</i> (Py-GC/MS)	VOCs from 6 nicotine salts	E-liquid nicotine salt	>90% of nicotine citrate/tartrate/malate is transferred to the smoke; pyrolysis products are predominantly aldehydes & acid anhydrides
14	(Zhang et al., 2019)	Simultaneous determination of 4 aldehydes by HS-GC-MS	HS-GC-MS + <i>isotope internal standard</i>	<i>Acetaldehyde, propionaldehyde, acrolein, crotonaldehyde</i>	Mainstream cigarette smoke	Does not require derivatization DNPH; $R^2 > 0.992$ ; LOD 0.014–0.12 $\mu\text{g/cigarette}$
15	(Zhao et al., 2023)	Health risk assessment of organics & heavy metals in e-cigarettes	Analytical review + risk assessment	<i>Formaldehyde, acetaldehyde, acrolein, acetone</i> + 7 logam berat	<i>E-liquid &amp; aerosol</i>	Inhalation risk is much higher than dermal/oral exposure; aldehydes contribute to significant carcinogenic risk.

A review of the 15 included articles revealed a wide diversity in the GC-MS instrumentation approaches applied to the determination of aldehydes and other hazardous volatile compounds in e-liquids and e-cigarette aerosols. The main GC-MS method variants identified include conventional GC-MS with various sample preparation techniques, headspace GC-MS (HS-GC-MS), solid-phase microextraction (SPME) GC-MS, pyrolysis GC-MS (Py-GC/MS), time-of-flight GC-MS (GC-TOF-MS), and the combination of GC-MS with ion mobility spectrometry (GCxIMS). This diversity reflects the lack of a single method that has been universally adopted as a standard for the analysis of aldehydes in e-liquids, which in turn presents a major challenge in data harmonization across laboratories (Dagla, 2023).

The HS-GC-MS approach is one of the most widely used variants because it offers a number of significant practical advantages. This method does not require a chemical derivatization step using the 2,4-dinitrophenylhydrazine (DNPH) reagent commonly used in HPLC-UV methods, thus avoiding potential interferences caused by derivatization reaction byproducts. (Baldovinos et al., 2024) demonstrated that the HS-GC-MS method with the addition of isotopic internal standards was able to simultaneously quantify four aldehydes—namely, acetaldehyde, propionaldehyde, acrolein, and crotonaldehyde—with excellent linearity ( $R^2 > 0.992$ ) without the need for derivatization, and with LOD values ranging from 0.014 to 0.12  $\mu\text{g}$  per cigarette. (Golpe et al., 2023) also developed a static headspace GC-MS method that is capable of characterizing VOCs in 146 e-liquid samples quantitatively and qualitatively without complicated sample preparation procedures, while also detecting the presence of benzene at concentrations that are of concern from a health perspective.

A more recent innovation was demonstrated by (Eshraghian & Al-delaimy, 2021) who introduced an aerosol collection system using refrigerated headspace vials, which were then analyzed by either HS-GC-MS or HS-GCxIMS. This system is designed to be widely implemented even by laboratories with limited budgets, as it uses standard equipment available in most analytical laboratories. The combined GC-MS and GCxIMS approach proved effective in identifying unknown compounds in complex aerosol matrices, including degradation products not yet listed in standard reference spectral libraries. Another interesting variant is pyrolysis-GC/MS, utilized by (Lebouf, 2018) to analyze the thermal products of six types of nicotine salts commonly used in e-liquids, where more than 90% of the nicotine in the citrate, tartrate, and malate salts is transferred to the aerosol along with various potentially toxic pyrolysis products. Meanwhile, (Krüsemann et al., 2020) applied GC-TOF-MS with a non-target screening approach to identify more than 250 compounds in commercial e-liquids simultaneously, resulting in a much more comprehensive identification rate than single-target methods.

Evaluation of method validation parameters reported in 15 studies revealed highly variable levels of completeness across studies, reflecting the lack of uniform validation standards in e-liquid analysis. The most frequently reported parameters included linearity ( $R^2$ ), limit of detection (LOD), limit of quantification (LOQ), percent recovery, and relative standard deviation (RSD). In terms of linearity, nearly all GC-MS methods studied performed very well, with  $R^2$  values consistently above 0.990. (Wei et al., 2021) reported  $R^2 > 0.9932$  for nine volatile compounds in cigarette adhesive matrices using HS-GC-MS, while (Zhao et al., 2023) also reported  $R^2 > 0.992$  for four aldehydes in mainstream cigarette smoke, demonstrating the ability of HS-GC-MS to maintain excellent linearity even in complex matrices.

In terms of sensitivity, reported LOD values show significant variation depending on sample preparation techniques and instrumentation conditions. Wei et al. (2021) reported LODs in the range of 3.1–147.7 ng/g, while Zhang et al., 2019 achieved LODs as low as 0.014  $\mu\text{g}$  per cigarette for acetaldehyde. Regarding accuracy and precision, most studies reported recovery percentages in the range of 70–115%, which generally meets the requirements of international validation guidelines such as ICH Q2(R1) and AOAC. The use of isotopic internal standards—as implemented by Yang et al., (2023) with an RSD below 10%—has been shown

to effectively compensate for matrix variations and instrument fluctuations, making it a highly recommended best practice, although not consistently implemented across the reviewed studies. Important methodological contribution by developing an on-fiber derivatization SPME technique that combines the sampling and derivatization stages in a single step, opening up the possibility of real-time dynamic measurement of carbonyl compounds during the vaping process.

Based on a synthesis of all studies, the most consistently detected aldehyde compounds were formaldehyde, acetaldehyde, and acrolein—all three of which are inherent thermal decomposition products formed from the main e-liquid components, propylene glycol (PG) and glycerol (VG), when exposed to heat from the device's heating coil. (Telgheder et al., 2023) provided strong experimental evidence that increasing the voltage from 3.3 V to 4.8 V caused the total aldehyde emission rate to triple, with acrolein increasing by up to 10-fold, and identified glycidol as a previously unreported probable carcinogen in e-cigarette aerosol. (Wang et al., 2024) furthered this understanding by detecting formaldehyde-hemiacetal in the aerosol of devices operating above 11.7 W, indicating that conventional measurements that capture only free aldehydes likely substantially underestimate the true total aldehyde exposure. (Ogunwale et al., 2017) complemented the toxicological picture by showing that exposure to acrolein and formaldehyde via inhalation resulted in risk estimates exceeding regulatory acceptance limits, confirming that the determination of aldehyde levels in e-liquids is an urgent need from a public health protection perspective.

A comparative analysis of all studies also revealed several substantial methodological gaps. The lack of uniform method standards poses a fundamental challenge, as each laboratory develops its own procedures, making objective comparisons between studies extremely difficult. Furthermore, most studies focus on aerosol analysis during the vaping process, while direct analysis of e-liquids before heating has received relatively little attention—despite their importance for understanding the raw material's contribution to total aldehyde exposure and for product quality control purposes prior to distribution. The majority of studies also used devices from a specific generation that may no longer be representative of currently available products, while the use of certified reference materials (CRMs) specific to e-liquid matrices remains very limited, leading to uncertainties in the metrological traceability of the resulting data.

Based on these findings, several critical methodological recommendations can be formulated. The development and adoption of HS-GC-MS reference methods with isotopic internal standards for the simultaneous determination of major aldehydes (formaldehyde, acetaldehyde, acrolein, crotonaldehyde) in e-liquids should be prioritized. All international validation parameters should be consistently reported in any new publications in this area. The identification and quantification of aldehydes in their bound form (hemiacetals) should be integrated into standard analytical protocols to provide more toxicologically accurate estimates of total exposure. Finally, international collaboration between laboratories to conduct interlaboratory comparison studies using the same reference e-liquid samples is urgently needed to assess the comparability and reproducibility of widely used GC-MS methods (Sala et al., 2017).

## CONCLUSION

A systematic review of 15 GC-MS-based studies confirmed that this method is a superior analytical platform for the determination of aldehydes in e-cigarette e-liquids, with isotopic internal standard headspace GC-MS being the most optimal variant in terms of validation performance. Formaldehyde, acetaldehyde, and acrolein were consistently identified as the primary thermal decomposition products of propylene glycol and glycerol, with their emission intensities positively correlated with device operational parameters such as voltage and power. The presence of formaldehyde-hemiacetal indicates substantial underestimation of

the conventional approach, making a comprehensive analytical protocol that includes the bound aldehyde fraction essential. Significant methodological fragmentation between laboratories, the absence of certified reference materials specific to e-liquid matrices, and the lack of validation on current-generation devices represent critical gaps that need to be addressed immediately. Therefore, harmonization of HS-GC-MS-based reference methods through international interlaboratory collaboration and the development of standardized regulatory standards is an urgent priority to ensure the accuracy of comprehensive and sustainable risk assessments of aldehyde exposure in e-cigarette users.

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