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Validated GC- FID Method for the Determination of Acetic Acid as an Impurity in Triacetin by Gas Chromatography

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ABSTRACT: A simple and robust gas chromatography method with flame ionization detection (GC-FID) was developed and validated for the determination of acetic acid as an impurity in triacetin, which is an important value added biorefinery product. The method utilized a DB-624 capillary column with programmed temperature and methanol as the diluent. The method was validated as per ICH Q2(R1) was evaluated for specificity, linearity, accuracy, precision, LOD, LOQ, robustness, and system suitability. The developed method showed a good linearity ($R^2 > 0.998$) having LOD and LOQ of 100 µg/mL and 300 µg/mL, respectively. The approach has successfully been used to follow acetic acid for samples from triacetin hydrolysis.

KEYWORDS: Triacetin, Acetic acid, GC-FID, Method validation

I. INTRODUCTION

Triacetin is glycerol triacetate. It is commercially applied in many biorefineries, pharmaceuticals, food, and cosmetic products and has good attributes as a plasticizer, humectants, and solvent. Triacetin is used in food (as a food additive), and in cosmetics, it has been known to be used for a variety of purposes. Used in drug formulation due to its chemical stability, low toxicity and good compatibility with different other excipients.

Acetic acid is a weak organic acid and an important ingredient for making triacetin. It is often used to acetylate glycerol to produce triacetin. If the purification process isn't thorough, some acetic acid may remain in the final product. Acetic acid is also used in pharmaceutical formulations to adjust pH, as a preservative, and as a reagent in analytical chemistry.

Measuring triacetin and acetic acid is crucial during raw material analysis, process monitoring, and testing the finished product. The purity of triacetin ensures that it has consistent plasticizing properties and stability in the dosage form. Monitoring the acetic acid content is important because there are regulatory limits on residual solvents or impurities that could impact product safety and effectiveness.

Several analytical techniques identify triacetin and acetic acid, including gas chromatography (GC), high-performance liquid chromatography (HPLC), and titrimetric methods. Among these, gas chromatography with flame ionization detection (GC-FID) is widely preferred because it has high sensitivity and specificity. It can also quantify volatile and semi-volatile components at the same time. For acetic acid determination, researchers either perform direct GC analysis or use derivatization methods to improve volatility and detection.

Developing and validating a strong analytical method for simultaneous estimation of triacetin and acetic acid is crucial. Its quality is affected by residual acetic acid from the synthesis, which needs precise quantification. Traditional titration methods do not have the sensitivity needed for detecting acetic acid. This study presents a validated GC-FID method for measuring acetic acid in a triacetin matrix. Meeting ICH guidelines (Q2(R1)) is essential. The validated method should show accuracy, precision, linearity, robustness, specificity, and sensitivity (LOD and LOQ) to ensure reliability in routine quality control.

In this study, we developed and validated a gas chromatographic method to measure triacetin and acetic acid in biorefineries preparations. The method allows for simple sample preparation, good reproducibility, and meets regulatory requirements. This enables its use in quality assurance and stability studies in manufacturing.

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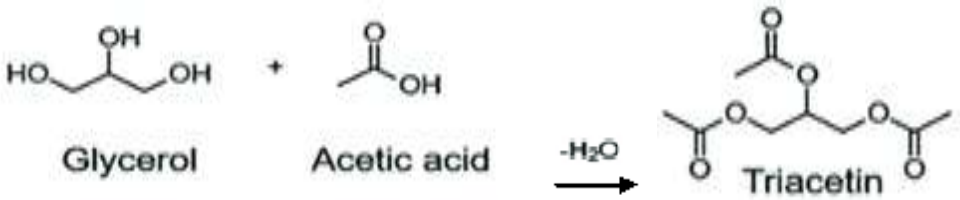
II. MATERIALS AND METHODS

We used analytical grade triacetin (glycerol triacetate) with purity greater than 99%. We also use Glacial acetic acid; Methanol (AR grade) was procured from Merck chemical. We filtered it through a 0.22 µm membrane filter to prepare the solution. All reagents were used without further purification, and prepared freshly to prevent contamination or degradation.



III. EXPERIMENTATION

GC Conditions: The analysis was performed using a gas chromatograph equipped with a flame ionization detector. The system was fitted with a capillary column (DB- 624, 30 m × 0.53 mm × 3 µm) and nitrogen of 99.99% purity was used as the carrier gas at a flow rate of 2.0 mL per minute. The injector temperature was maintained at 250°C and the detector temperature at 300°C. The column oven temperature program was initiated at 100°C with a hold time of 0.5 minutes, followed by a ramp of 10°C per minute up to 220°C with a final hold of five minutes, total run time is 28 minutes, Injection volume is injected as 0.6 µL, split ratio 1:50, signals used for the acquisition with the 50Hz/0.004min. Data acquisition and integration were carried out using the Agilent manufacturer-Application-EZChrom- OpenLAB 2.1(2.1.0) chromatographic software.

Standard Preparation: A stock solution of triacetin at a concentration of 1000 ppm, 50 mg of triacetin was accurately weighed into a 50 mL volumetric flask and diluted to the mark with methanol. Similarly, 0.286 gm of glacial acetic acid was pipette into a 100 mL volumetric flask and was methanol diluted to achieve a stock concentration of 500 ppm of acetic acid. The stock solutions were serial diluted to cover the required range of concentrations for acetic acid such as 100, 300, and up to 1000 ppm to provide working standard solutions for flow-through tests and system suitability tests. The solution was filtered through a 0.22 µm PTFE membrane filter prior to gas chromatographic analysis. Spiked samples for the precision and recovery study were prepared using a triacetin matrix.

Sample Preparation: For sample preparation, a quantity of sample equivalent to approximately 100 mg of triacetin was weighed accurately and transferred to a 100 mL volumetric flask. Fifty milliliters of methanol were added, and the mixture was sonicated for two minutes to ensure complete extraction. After cooling, the volume was made up to the mark with methanol, mixed well, and filtered through a 0.22 µm membrane filter before injection into the chromatograph.

Method Validation: The method was validated according to the ICH Q2 (R1) guidelines. The system suitability was evaluated by six replica injections of the standard solution and evaluated for standard deviations relative to the peak region and perception time stability. Lineiness was established in five concentration levels covering 70 to 130% of the target concentration by plotting concentration against the peak field and calculating the regression equation and correlation coefficients. The accuracy was evaluated by recovery studies on three levels (50%, 100%, and 150%), which was through standard extra for pre-analyzed samples, and percentage recovery and relative standard deviations were calculated. Analysis was determined through repetition (intra-day) by analyzing the same day and six replications at 100% concentration on the same day and intermediate procedure (inter-day) by analyzing different days or different analysts. The retention of triacetin and acetic acid was confirmed by analyzing the blank, placebo, standard and sample solutions to no interference on time. The range of detection and permutation was determined on the basis of 3: 1 and 10: 1 signal-to-shape ratio or using the standard deviation of the response and slope method. Strengthening was evaluated by deliberately separate chromatographic parameters carrier gas flow from ± 0.2 mL per minute, and the system suitability and impact on the assay results were assessed.

IV. RESULTS AND DISCUSSION

Chromatographic Separation - Chromatographic analysis of the developed method demonstrated effective resolution between acetic acid and triacetin under optimized gas chromatographic conditions.

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The retention time of acetic acid was observed at about 4.8 minutes, while the triacetin was elected at about 15.3 minutes. Both peaks were well solved with no interference from blank, excipients, or other components, confirming the specificity of the method.

The peaks obtained for both analyzes with tail factors within the acceptable boundaries (<2.0) according to the system suitability criteria were sharp, symmetrical and well defined. The baseline remained stable in the entire analysis, indicating the absence of co-eluting peaks or matrix intervention. The resolution between acetic acid and triacetin peaks exceeded the minimum requirement (R> 2.0), confirming that chromatographic conditions were suitable for simultaneous finance.

Customized temperature programs and stable phase provided copyable retention time, in which retention of retention time below 2.0 % in repeating injections reflects the strength of the RSD method. These results ensure sample metrics such as capsules shell formulations or a reliable identity and stagnation of quantity of both acetic acid and triacetin in wholesale exercises.

Linearity: The linearity of the gas chromatographic method developed for triacetin was evaluated at the range of 350–650 µg/ml. A series of calibration standards were prepared at five concentration levels to suit 70%, 80%, 100%, 120%, and 130%of the target concentration. Each level was analyzed in the duplicate, and the average peak area was used for calibration curve manufacture.

A calibration curve was plotted with concentration (µg/ml) on X-axis and peak area on the Y axis. The data exhibited a strong linear relation with the regression equation as below-

$y=307.8 X +85772$ ……(1)

Where the Y represents the peak field and represents the concentration of triacetin in X/mL Correlation coefficient (R^2 = 0.9989) confirmed the outstanding linearity's in the tested range, completing the ICH acceptance criteria (R^2> 0.99).

LINEARITY LEVEL	Corrected Concentration (ppm)	Acetic Acid (Area)
70%	368.8	27416
80%	421.4	44540
100%	526.8	76682
120%	632.2	107147
130%	684.8	126230
	Slope	307.8
	Y intercept	85772
	Correlation Coefficient	0.999

Table 1: Calibration Data and Linearity Results

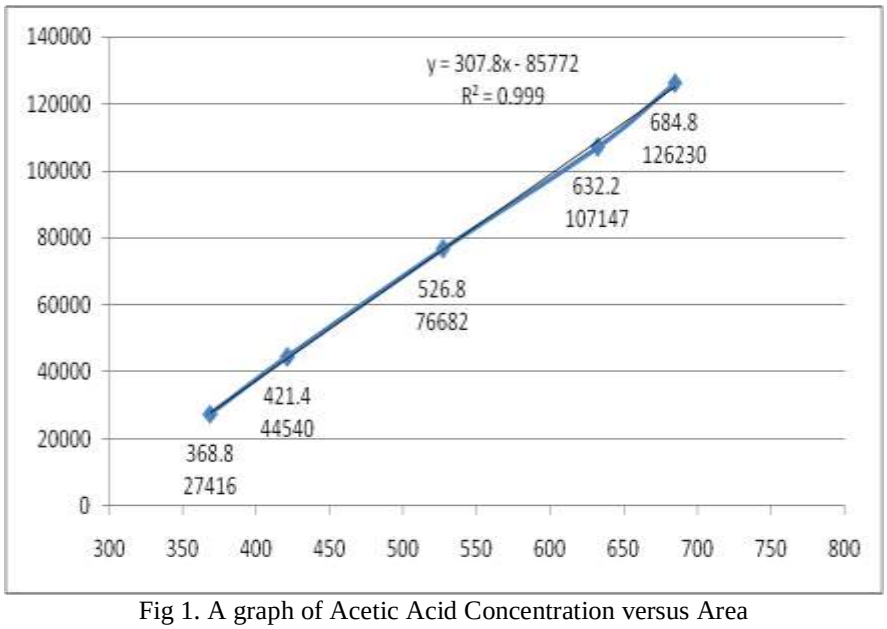

Fig 1. A graph of Acetic Acid Concentration versus Area

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The calculated slope of the calibration curve was 307.8, with an intercept of 85.772. Correlation coefficient (R^2) was found to be 0.999, which displays excellent linearity's and indicates that the method is suitable for the stagnation of the exact amount of triacetin within the specified range.

Sensitivity - LOD & LOQ: The limit of detection (LOD) and quantification (LOQ) for acetic acid was established based on the signal-to-noise approach and confirmed through practical analysis of diluted standard solutions. A series of progressive low concentrations of acetic acid solutions were prepared by the serial dilutions of the stock solution and injected into the gas chromatograph. The lowest concentration at which the analysis can be firmly detected (signal-to-show ratio ~ 3: 1) was considered LOD, while the lowest concentration that could be quantified with acceptable accuracy and precision (signal to noise ratio 10:1) was designated as the LOQ. During experimental determination, 100 ppm concentration was identified as LOD and 300 ppm concentration in the form of LOQ for acetic acid under the developed method conditions.

Limit of detection (LOD): The LOD solution (100 ppm) was injected into triplicate to verify peak detectability and assess the system repeatability at this level. The area of peak obtained as below-

Inj No.	Acetic acid Area
1	7329
2	7413
3	7421
Average	7388
Std. Dev	50.96
RSD %	0.69

The average area was calculated as 7388, with a standard deviation (SD) of 50.96 and a relative standard deviation (RSD) of 0.69%, confirming consistent response at the LOD level.

Limit of Quantification (LOQ) and Precision: For LOQ evaluation, 300 ppm solutions were injected three times to assess accuracy in this concentration. The received chromatograms were recorded, and the results are summarized below-

Inj No.	Acetic acid (Area)
1	34789
2	34282
3	34947
Average	34673
Std. Dev	347.43
RSD %	1.00

The average area was 34,673, with a standard deviation (SD) of 347.43 and RSD of 1.00%, within the acceptable range (<2%), indicating good precision at the LOQ level. Results show that the developed method is capable of detecting acetic acid at 100 ppm and determines it accurately at 300 ppm, meeting sensitivity

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requirements for monitoring impurities in biorefineries quality control.

The accuracy of the developed gas chromatographic method was evaluated in the context of ICH Q2 (R1) Guidelines after recurrence (intra-day procedure) and intermediate procedure (inter-day accurate).

Precision: The precision of the developed gas chromatographic method was evaluated in the ICH Q2 (R1) Guidelines after repeatability (intra-day procedure) and intermediate procedure (inter-day precision).

Repeatability: Six replication injections of acetic acid 500 ppm solution (5 mL in 1 gram matrix) were prepared and analyzed in the same chromatographic conditions using the same tool and analyst in a single day. The individual peak area obtained for these replicate injections was consistent, with an average peak area of 144,974.3, with a standard deviation of 2,531.24 and a relative standard deviation (RSD) of 1.75%. It displays excellent recurrence or repeatability of low RSD value method.

Inj No.	Acetic acid Area
1	147971
2	147628
3	146540
4	142571
5	142032
6	143104
Average	144974.3
Std. Dev	2531.24
RSD %	1.75

Intermediate precision: The intermediate procedure was evaluated by conducting analysis deliberately in various conditions, including different analysts on different days. Six replicates injections of acetic acid spiked at the 100% concentration level were prepared and analyzed. The average peak area received under these conditions was 118,072.5, with a standard deviation of 2,098.33 and RSD of 1.78%. These results confirm that this method still maintains high precision, when important variables such as analysts, day and equipment change.

Inj No.	Acetic acid Area
1	119233
2	115502
3	121012
4	118282
5	118593
6	115813

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Average	118072.50
Std. Dev	2098.33
RSD %	1.78

The RSD values for both repeatability (1.75%) and intermediate precision (1.78%) are well within the acceptance criteria of ≤2% for quantitative methods, confirming that the method is precise and suitable for routine quality control analysis of acetic acid in the presence of triacetin.

Accuracy / Recovery: The accuracy of the developed method was evaluated by recommending 50%, 100%and 150% of the test concentration of acetic acid, by the evaluation of the evaluation of the developed method. The known volume of acetic acid was pointed in matrix (triacetin) at these levels, and each pointed level was analyzed in three copies under customized chromatographic conditions. For each concentration level, percentage recovery was calculated using the following formula:

$$\text{Recovery (\%)} = \frac{\text{Added amount}}{\text{Received amount}} \times 100$$

Where the amount received was taken from the linear regression equation of the calibration curve:

$$\text{Received amount (µg/ml)} = \frac{\text{Area - Intercept}}{\text{Slope}} \quad ..(3)$$

The calibration curve created for recovery studies demonstrated excellent linearities in a concentration range of 50% -150% with regression equation:

$y = 1732x - 25307$ ….4)

and a correlation coefficient of 0.999 (R^2), which indicates a strong linear relationship between the peak area and concentration.

At 50% level, the average area was 62,179.00, with a standard deviation of 978.67 and a relative standard deviation of 1.57% (RSD). The recovery calculated at this stage was 99.977%. At 100% level, the average area was 146,132.67, with SD of 1,490.51 and RSD of 1.02%, which led to a recovery of 99.990%. At 150% level, the average area was 235,385.00, with SD of 4,007.86 and RSD of 1.70%, resulting in a recovery of 99.994%.

Level (%)	Replicate (Areas)	Mean (Area)	SD	RSD (%)	Recovery (%)
50%	61917	62179.00	978.67	1.57	99.977
	63262				
	61358				
100%	147456	146132.67	1490.51	1.02	99.990
	146424				
	144518				
150%	234961	235385.00	4007.86	1.70	99.994
	231606				
	239588				

Table. Accuracy with 50%, 100%, 150% concentration with Area obtained and Recovery.

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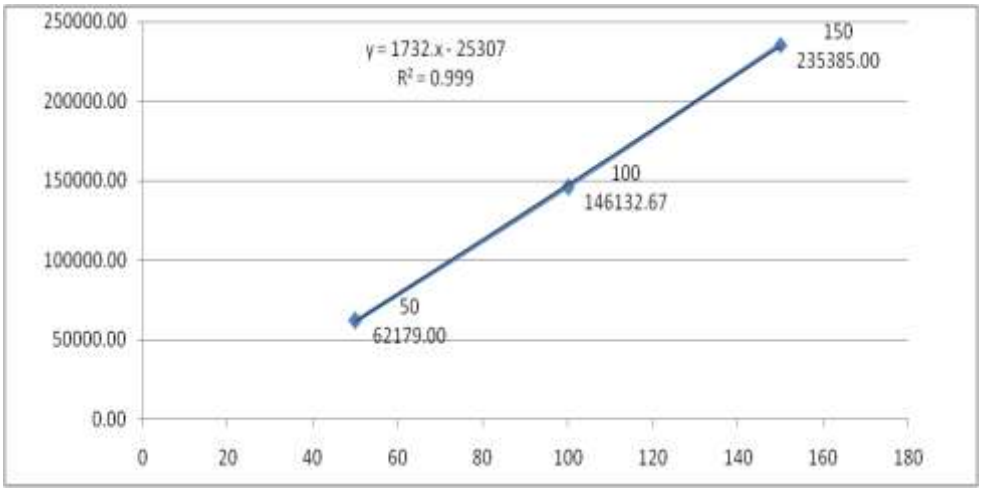
CONC %	AREA
50	62179.00
100	146132.67
150	235385.00



Used the formula:
Obtained Conc. = (Area – Intercept) / Slope
Recovery = (Obtained / Added) × 100

Observations: These recovery values, within 98–102% acceptance with low RSD values (<2%), confirm that the method is highly accurate and suitable for the quantification of acetic acid on the concentration range. The high correlation coefficient supports the reliability of further calibration models used for the quantification.
Precision for triplicate injections % RSD ≤ 2% is acceptable.
Linearity across level is good $R^2 = 0.999$
Robustness: The robustness of the developed gas chromatographic method was evaluated to determine the reliability of the method under deliberate variation in chromatographic conditions [1]. This study ensures that minor changes in method parameters do not significantly affect analytical results or system suitability norms.
One of the major parameters assessed was the flow rate of carrier gas. The standard flow rate used for analysis was 2.0 mL/min. To assess the robustness, the flow rate was different from ± 0.2 mL/min, resulting in two additional conditions: a low flow rate of 1.8 mL/min and increased flow rate of 2.2 mL/min.
Procedure: For each change in the operation condition of method, a standard solution of acetic acid (500 ppm) was injected six times. Retention time and peak area were recorded for each injection, and mean, standard deviation (SD), and relative standard deviation (% RSD) was calculated to evaluate precision under stressful conditions.

Flow Decreased (1.8 mL/min)

Injection	Retention time	Area
1	5.245	135210
2	5.235	133021
3	5.244	135367

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4	5.244	137098
5	5.243	136691
6	5.245	133037
Mean	----	135070.67
----	----	1742.40
RSD %	----	1.29

Flow Increased (2.2 mL/min)

Injection	Retention time	Area
1	4.459	107345
2	4.47	104897
3	4.454	105390
4	4.464	106698
5	4.459	106558
6	4.465	102048
Mean	----	105489.33
----	----	1910.49
RSD %	----	1.81

At the rate of 1.8 mL/min, retention time was 5.24 minutes on an average, and the average peak area was 135,070.67, with a standard deviation of 1,742.40 and %RSD of 1.29 %. Similarly, at an increased flow rate of 2.2 mL/min, the retention time was an average of 4.46 minutes, and the average peak area was 105,489.33, with a standard deviation of 1,910.49 and a %RSD of 1.81 %. Both sets of results satisfied predetermined acceptance criteria, where % RSD should not exceed 2 % and do not cause any significant peak deformation or loss of resolution. Although the approximate changes in retention time were seen with changes in flow rate (retention and low retention in increased flow when prolonged and reduced retention), peak shape, resolution and quantitative remained unaffected. These conclusions confirm that the method is robust for small changes in the carrier gas flow rate and suitable for routine application in quality control laboratories.

System Suitability: The system suitability testing was performed to ensure that the gas chromatographic system was performing adequately before and during analysis. A standard solution of acetic acid (500 ppm) was injected five consecutive times under optimized chromatographic conditions. The peak areas were recorded for each injection, and average, standard deviation (SD) and relative standard deviation (% RSD) were calculated to assess the accuracy and suitability of the system for analysis.

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Inj No.	Acetic acid (Area)
1	104353
2	106190
3	108027
4	107939
5	105421
Average	106386.00
Std. Dev	1597.45
RSD %	1.50

The average peak area received for five replica injections was 106,386.00, with a standard deviation of 1,597.45 and a %of 1.50 %RSD. This value is well within the acceptance criteria of the system suitability guidelines. 2.0% RSD. Additionally, there was no significant variation in retention time or peak symmetry during injections, confirming that the analytical system was stable and continuously performing.

Specificity: The specificity of the method was evaluated to ensure its ability to unevenly assess acetic acid in the presence of other potential matrix components including triacetin and excipients. To display specificity, the following solutions were individually analyzed: blank (Diluent- Methanol), the standard solution of acetic acid (500 ppm), triacetin as matrix, and spiked the acetic acid solutions in the matrix at 50%, 100% and 150% level samples.

Chromatograms obtained for the blank and triacetin matrix showed no peaks on retention time of acetic acid (about 4.8 minutes), which confirm the absence of interference. The standard acetic acid solution produced sharp, symmetric peaks at the required retention time, and the spiked samples demonstrated the same retention characteristics with proportional growth in the peak area corresponding to the level of concentration. These observations confirm that the method is specific for the determination of acetic acid even in the presence and excipients components.

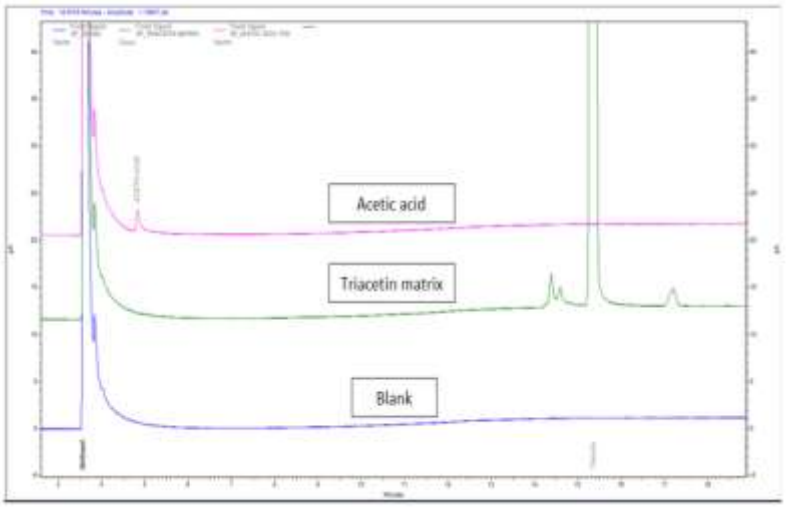

Fig . Overlay with stack of Blank, Triacetin and Acetic acid chromatograms.

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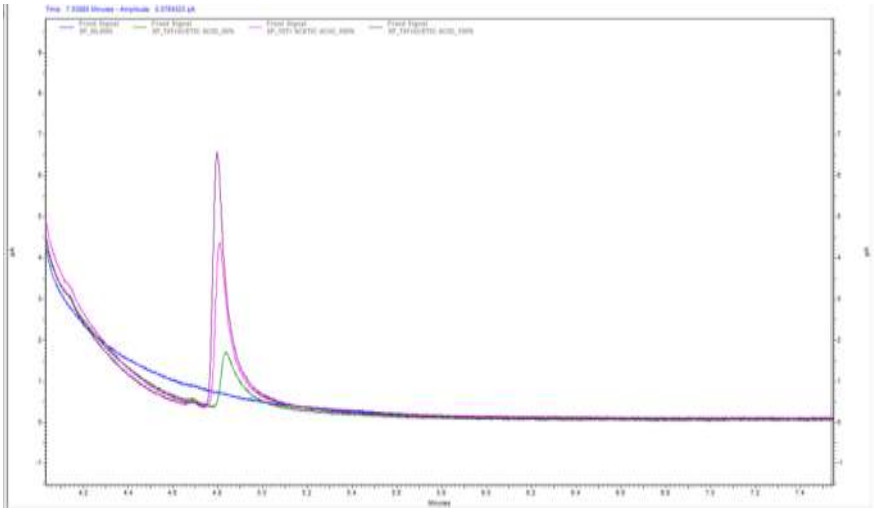

Fig . Overlay of Blank, 50%, 100% and 150% level Acetic acid chromatograms.

An analysis of the blank solution (diluents) detected no detectable peaks on the retention time of acetic acid, which confirms the absence of interference from the solvent system. Similarly, the chromatogram of the triacetin matrix showed no peaks on retention time corresponding to acetic acid, indicating that the matrix does not contribute to the interfering signals.

The injection of acetic acid standard solution (500 ppm) produced a clear, sharp peak on the expected retention time (~ 4.8 min), which validates the identification of analysis under optimized chromatographic conditions. In addition, spikes containing acetic acid at the level of 50%, 100%, and 150%concentration in triacetin matrix perform well -solved peaks on the same retention time, with proportional to spiked concentrations with peak areas. No additional peaks were observed in these chromatograms, which confirm the uniqueness of the method for acetic acid determination in the presence of triacetin and excipients.

V. CONCLUSION

A gas chromatographic method (GC) for the quantification of acetic acid with flame ionization was successfully developed in the presence of triacetin and successfully developed. The method provides effective separation of acetic acid and triacetin peaks, retention time of about 4.8 minutes and 15.3 minutes respectively, and displays sharp, symmetrical peaks without interference from blank, matrix or excipients.

The developed method was strictly valid for analytical method verification as per the ICH Q2 (R1) guidelines. Major verification parameters, including linearity, accuracy, accuracy, specificity, specificity and system suitability were systematically evaluated. The method demonstrated over 0.999 correlation coefficients, high recovery rate within the range of 98–102%, and a correlation coefficient with a correlation coefficient with low %RSD value (<2%) for both repeatability and intermediate precision, confirmed its reliability for quantitative analysis. Robustness studies have shown that intentional variation in chromatographic conditions, such as carrier gas flow rate, did not significantly affect the results of system suitability or quantitation results, and support method reliability under more regular laboratory conditions.

Overall, this valid GC- FID method is suitable for regular quality control analysis of acetic acid in triacetin-based samples or raw materials, ensures compliance with regulatory specifications and supports the quality of the product continuously in biorefineries, drugs and related applications.

SOME OF THE ADVANAGES FROM THE ABOVE RESULTS

a) Fast & accurately determination of acetic acid from triacetin.
b) Robust method
c) Effective separation of acetic acid and triacetin peaks.

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