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Case Report

Proficiency Testing Program for Fly Ash: Ensuring Quality and Consistency Through Standardized Evaluation

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Abstract

This case study evaluates a Proficiency Testing Program (PTP) for fly ash testing, focusing on the homogeneity, stability, and performance of participating laboratories. The study aims to identify areas for improvement in testing methodologies to enhance the reliability of fly ash analysis. The PTP adheres to international standards, including ISO 13528:2015 and ISO/IEC 17043:2010, for sample preparation, testing, and data analysis. The PTP ensures the homogeneity of test samples through meticulous division, testing, and data-driven analysis. The stability of samples is monitored through regular tests at predetermined intervals, with results compared to initial homogeneity tests. The methodology of evaluation involves calculating Z' (z-prime) scores to assess the performance of participant laboratories, with scores within ±2.0 considered satisfactory and scores beyond ±3.0 considered outliers. The results of the proficiency testing reveal variations in the performance of participant laboratories across specific gravity, fineness, compressive strength at 7 days, and soundness by autoclave parameters. While most laboratories demonstrate adherence to expected values, some deviations raise concerns and highlight the need for continuous improvement. Laboratories with deviations are encouraged to evaluate their testing methodologies and address factors contributing to the observed deviations. Overall, the study underscores the importance of proficiency testing in ensuring the quality and consistency of fly ash testing. Continued development and refinement of proficiency testing programs are essential for the sustainable utilization of fly ash in various applications.

Keywords

Fly Ash, Proficiency Testing, Quality Assurance, Construction Materials, Standardization

1. Introduction

Proficiency testing programs (PTPs) play a crucial role in ensuring the quality and reliability of laboratory testing results, particularly in industries like construction materials where accuracy is paramount. In this research paper, our specific objectives are to evaluate the effectiveness of existing PTPs in assessing the proficiency of laboratories involved in fly ash testing and to propose enhancements for optimizing their utility in this context.

By examining the current landscape of PTPs for fly ash testing, we aim to identify strengths, weaknesses, and areas for improvement. Through a comprehensive analysis, we seek to provide insights that can inform the development of more robust proficiency testing frameworks tailored to the unique characteristics of fly ash analysis.

This research endeavors to contribute to the advancement of quality assurance practices in the construction materials

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industry, ultimately enhancing the reliability of fly ash testing results. Through rigorous investigation and evidence-based recommendations, we aspire to support stakeholders in implementing effective strategies for maintaining the integrity and accuracy of laboratory testing in this critical domain.

In the ever-evolving landscape of sustainable construction and resource utilization, fly ash, a by-product of coal combustion, emerges as a material brimming with potential. This finely divided powder, primarily composed of silicon dioxide, aluminium oxide, and iron oxide, boasts a spectrum of valuable properties that translate into diverse applications across various industries. From serving as a key ingredient in high-performance concrete to enhancing geotechnical engineering projects, promoting agricultural productivity, and contributing to waste stabilization, fly ash offers an environmentally friendly and economically attractive alternative to traditional materials.

However, harnessing the full potential of fly ash hinges on one crucial aspect: consistent and accurate quality assessment. Due to variations in coal source and combustion conditions, fly ash properties can exhibit significant differences. Ensuring its suitability and safety for specific applications necessitates standardized, reliable testing procedures. This is where proficiency testing programs (PTPs) play a vital role.

PTPs serve as a controlled environment for laboratories conducting fly ash testing to assess their analytical capabilities and compare their performance with their peers. In the past, various PTPs have been conducted for different products including construction materials [1-8]. By participating in such programs, laboratories gain valuable into their strengths and weaknesses, ultimately leading to continuous improvement in their testing practices. Additionally, PTPs contribute significantly to the overall quality and consistency of fly ash testing and promote confidence in their use across various sectors. This case study delves into the analysis of a specific PTP designed to evaluate the proficiency of participating laboratories in testing key parameters of fly ash. Utilizing established test methods as per IS 1727:1967 [9] or specific gravity, fineness, compressive strength (7 days), and soundness by autoclave, the PTP aimed to provide a comprehensive assessment of the laboratories' ability to accurately and consistently analyze this versatile material.

By examining the design, procedures, and results of this PTP, we gain valuable insights into the effectiveness of standardized evaluation methods in ensuring the quality and reliability of fly ash testing. Moreover, the research identifies areas for improvement and potential avenues for further exploration, ultimately contributing to the advancement of responsible and sustainable fly ash utilization in a diverse range of applications.

2. PT Item Preparation

To ensure the safe and responsible use of fly ash, it is crucial to maintain consistency and accuracy in its testing. The PTP paid close attention to established standards when preparing its test samples. Both the fly ash and cement were sourced according to Indian Standards IS 3812 (Part-1):2013 [10] for Fly ash and IS 269:2015 [11] for OPC-53 grade, and were subjected to rigorous homogenization processes. These processes were based on Clause 9.1 of IS 6491:1986 [12] for fly ash and IS 3535:1986 [13], respectively, to ensure uniformity within each material. Representative samples were then taken for further testing of key properties such as consistency, density, and compressive strength. The remaining homogenized material was meticulously mixed and used to prepare the final PT items that were distributed to participating laboratories. By adhering to these high standards, the PTP ensured that all participants received consistent and reliable test materials, which facilitated a meaningful evaluation of their fly ash testing proficiency.

3. Homogeneity of PT Item

In any Proficiency Testing Program (PTP), the importance of ensuring homogeneity within test samples cannot be overstated. This entails guaranteeing uniformity across each sample, so that all participants work with identical materials, regardless of their location or testing order. This passage delves into the extensive measures taken to attain this critical aspect in a PTP involving cement and fly ash.

To begin, the organizers commenced with a 10kg cement sample, meticulously dividing it into two equal parts: 4kg and 6kg. The larger portion was then further subdivided into two equivalent parts for conducting duplicate compressive strength tests. Moreover, 2kg of fly ash was set aside for the 7-day Compressive strength test of fly ash. These divisions ensured an adequate amount of material for testing while maintaining consistency.

Ensuring consistency within batches was not sufficient. To verify true homogeneity, a dedicated testing process was implemented. Ten random samples of both cement and fly ash were carefully selected for analysis of specific gravity, soundness, and fineness. Each sample underwent duplicate testing, resulting in a total of 20 results per parameter. This meticulous approach provided a robust dataset for analysis.

To evaluate uniformity, the organizers calculated the between-sample standard deviation (SD) for each test parameter. This statistic captures the variability between different samples, indicating any potential inconsistencies. These calculated SDs were then compared to a pre-established criterion – a maximum of 0.3 times the predetermined standard deviation for each parameter (SDPA). This criterion served as a benchmark for acceptable homogeneity.

Ultimately, the selection of SDPAs was initially guided by expert judgment and past experience. However, the organizers recognized the importance of data-driven confirmation and took an additional step. They employed the standard deviations calculated from the participants' own test results, effectively verifying the homogeneity of the samples through an independent dataset. This additional step bolstered confidence in the consistency of the samples.

The comprehensive approach detailed in this description underscores the dedication to ensuring homogeneity in the PTP. Through diligent division, exhaustive testing with duplicate measurements, and data-driven analysis, the PT provider established a robust and dependable testing environment for all participants. This, in turn, enhances the overall efficacy and value of the Proficiency Testing Program.

Table 1. *Assessment of homogeneity.*

Parameters	Average	Between Sample Standard Deviation (S_s)	Limiting Value ≤0.3*SDPA
Fineness, m^2/kg	411.53	1.261	6.000
Specific Gravity, g/cc	2.71	0.003	0.030
Soundness by Autoclave, %	0.01	0.00	0.015
Average Compressive strength 7 days duration, %	87.01	0.00	1.50

Table 1 data suggests a high level of homogeneity across the evaluated parameters. The low standard deviations for all the parameters indicate a consistent and uniform quality in the samples. The observed values are within the specified limits, indicating that the measurements align with the desired standards.

4. Stability of PT Item

Ensuring the uniformity of samples within themselves (homogeneity) is just as important as their stability over time for the success of a Proficiency Testing Program (PTP). To guarantee the stability of cement and fly ash samples throughout the testing period, meticulous procedures are implemented. Regular stability tests are conducted at predetermined intervals, emphasizing continuous monitoring. These tests encompass three random samples for each parameter, including specific gravity, soundness, and fineness, demonstrating a dedication to thorough analysis. To further strengthen the data set, each sample undergoes double testing, resulting in six results per parameter for comprehensive evaluation.

The PT provider compared stability test results with the initial homogeneity test. A strict criterion ensured minimal acceptable change, protecting sample integrity. They also used participants' results for data-driven confirmation. This approach ensures a reliable and fair testing environment.

Table 2. *Assessment of Stability.*

Parameters	*Days*	*Average of stability Test*	*Average of homogeneity*	*Difference*	Limiting Value ≤0.3*SDPA
Fineness, m^2/kg	1st Day	412.00	411.53	0.467	5.437
	3rd Days	412.50		0.967	
	7th Days	411.00		0.533	
Specific Gravity, g/cc	1st Day	2.70	2.71	0.01	0.012
	3rd Days	2.70		0.01	
	7th Days	2.70		0.01	
Soundness by Autoclave, %	1st Day	0.01	0.01	0.00	0.005
	3rd Days	0.01		0.00	
	7th Days	0.01		0.00	
Average Compressive Strength 7 days duration, %	1st Day	87.44	87.01	0.428	0.813
	3rd Days	87.67		0.660	
	7th Days	87.48		0.473	

The analysis of the stability data shown in Table 2 reveals important insights about the tested parameters over the course of the evaluation. Regarding the fineness, there were slight variations observed throughout the 1st, 3rd, and 7th days of the testing schedules given to the participants laboratory, with differences ranging from 0.467 to 0.967. However, these differences were all well within the predetermined limiting value of ≤0.3 times the standard deviation for each parameter (SDPA), which was set at 5.437. Therefore, the fineness remained relatively stable and consistent throughout the testing period.

The specific gravity values remained almost unchanged from the 1st to the 7th day, with differences of only 0.01 observed. These differences were well below the limiting value of 0.012, indicating that the specific gravity exhibited excellent stability and consistency throughout the duration of the assessment.

For the soundness by autoclave parameter, the values remained constant at 0.01 throughout all three test intervals. As the observed differences were all zero, within the limiting value of 0.005, it can be concluded that the soundness by autoclave remained highly stable and consistent, showing no significant alterations that could affect the test results.

Assessing the average compressive strength over a 7-day period, there were minor differences observed, ranging from 0.428 to 0.660. Again, all these differences fell within the limiting value of 0.813, indicating that the average compressive strength experienced acceptable and expected variations, which did not compromise the reliability of the test results.

The stability data analysis demonstrates that the tested parameters exhibited a high level of stability and consistency throughout the assessment. The observed differences were minimal and well within the predetermined limits, ensuring the integrity and reliability of the test samples. Such meticulous attention to sample stability analysis guarantees a robust and dependable testing environment, contributing to the overall fairness and effectiveness of the program.

5. Methodology

The PTP adhered to internationally recognized standards, specifically ISO 13528:2015 [14] and ISO 17043:2010 [15] for sample preparation, testing, and subsequent data analysis. The program ensured the homogeneity and stability of the samples, and the performance of the participating laboratories was assessed using robust Z-scores.

5.1. Analysis of Data

The Assigned value and the uncertainty of the assigned value are evaluated by the robust analysis as per ISO 13528:2015. The participant's results are having metrological traceability and are in SI units. The performance of laboratories is evaluated by comparison with other participants. Initially the SDPA is chosen by perception. However, the acceptability of Homogeneity and Stability results are checked with SDPA from the participant's results. The trueness of the assigned value is verified as per Clause 7.8 of ISO 13528:2015. The results are found suitable and satisfactory.

5.2. Z′ Score (Z-prime) Evaluation

The results are evaluated as per ISO/IEC 17043:2010 and Clause 9.5 of ISO 13528:2015 by calculating the Robust Z′ Score of the test parameter of each participant laboratory. The Z′ prime score is considered as the uncertainty of assigned value is more than the stipulated criteria $u(x_{pt}) > 0.3\sigma_{pt}$ for all parameters.

The Z′ score is calculated as follows;

$$Z' = (x_i - x_{pt}) / \sqrt{\sigma^2_{pt} + u^2(x_{pt})}$$

where, x_i is the test result from participant laboratory, x_{pt} is the assigned value and σ_{pt} is the standard deviation for proficiency assessment (SDPA).

$$u(x_{pt}) = 1.25 s^* / \sqrt{p}$$

Where, $u(x_{pt})$ is the uncertainty of assigned values, s* is robust standard deviation and p is number of participants

As per ISO 13528:2015, performance of the laboratories with $|Z'| \leq 2.0$ is considered satisfactory. The laboratories getting $|Z'| \geq 3.0$ are considered outlier and those getting $2.0 < |Z'| < 3.0$ score are considered as questionable performers.

5.3. Statistical Findings

Seven results for Specific gravity & Compressive Strength and Six results for Fineness & Soundness by Autoclave have been statistically evaluated and Z'-scores is calculated. The details are given in Table 3.

Table 3. *Statistical Findings.*

Parameter	Specific Gravity, g/cc	Fineness, m^2/kg	Compressive strength-7d duration, %	Soundness by Autoclave, %
No. of Labs. (N) considered for statistical analysis	7	6	7	6

Parameter	Specific Gravity, g/cc	Fineness, m^2/kg	Compressive strength-7d duration, %	Soundness by Auto-clave, %
Minimum	2.66	394	86	0.00
Maximum	2.75	433	92	0.04
Average	2.697	411.7	89	0.013
Assigned Value	2.697	411.667	89.0	0.012
SDPA	0.035	16.144	2.450	0.015
Uncertainty of Assigned Value	0.016	8.238	1.157	0.008

The data shows valuable insights into the uncertainties associated with the assigned values of different parameters. Uncertainty plays a significant role in scientific measurements and is crucial for understanding the reliability and accuracy of the obtained results.

The uncertainties of the assigned values are represented by the values given in the "Uncertainty of Assigned Value" column. These uncertainties quantify the range within which the true value of each parameter is expected to lie, considering various sources of error and variability in the measurement process.

Analysing the specific gravity parameter, the assigned value of 2.697 g/cc, with an uncertainty of 0.016 g/cc, signifies that the true value is likely to fall within the range of 2.681 g/cc to 2.713 g/cc. This uncertainty range provides a measure of confidence regarding the true value and acknowledges potential variations introduced by measurement techniques, equipment limitations, and inherent variability in the material being tested.

Similarly, for the fineness parameter, the assigned value of 411.667 m^2/kg, with an uncertainty of 8.238 m^2/kg, reflects that the true value is expected to be within the interval of 403.429 m^2/kg to 419.905 m^2/kg. This uncertainty range accounts for potential errors in sample preparation, measurement methodology, and the inherent heterogeneity of the material being analysed.

Considering compressive strength with a 7-day duration, the assigned value of 89.0%, with an uncertainty of 1.157%, indicates that the true value is likely to fall within the range of 87.843% to 90.157%. This uncertainty range considers variations arising from factors such as specimen preparation, testing conditions, and the inherent variability in the strength properties of the material.

Lastly, for the soundness by autoclave parameter, the assigned value of 0.012%, with an uncertainty of 0.008%, suggests that the true value is expected to lie within the range of 0.004% to 0.020%. This uncertainty range acknowledges potential deviations caused by experimental procedures, equipment precision, and inherent variations in the material's resistance to autoclave-induced expansion.

By providing these uncertainties, the data promotes transparency and allows researchers, practitioners, and decision-makers to account for and consider the potential variations in the measured values. Moreover, it highlights the importance of uncertainty analysis in ensuring the robustness and validity of scientific findings, contributing to the overall credibility and reliability of research outcomes. Researchers can utilize this information to appropriately interpret and draw meaningful conclusions from the measured data while accounting for the inherent uncertainties in the assigned values.

6. Methodology of Evaluation

The test results which are received are analysed. The Participant Laboratories are uniquely coded by alphabets between ‘A’ to ‘G’. The results are evaluated as per ISO 17043:2010 and ISO 13528:2015. In case of specific gravity and compressive strength tests, seven participant’s results are considered for analysis. In case of fineness and soundness by autoclave tests, six participant’s results are considered for analysis.

The questionable performance and outliers in the Z’ score are given in the Table 4.

Table 4*. Questionable Performance.*

Parameter	N	No. of Questionable performance ($2<\|Z\|<3$)	No. of Outlying performance ($\|Z\|\geq 3$)
Specific Gravity, g/cc	7	Nil	Nil
Fineness, m2/kg	6	Nil	Nil

Parameter	N	No. of Questionable performance (2<|Z|<3)	No. of Outlying performance (|Z|≥3)
Compressive strength 7-d, %	7	Nil	Nil
Soundness by Autoclave, %	6	Nil	Nil

The data in Table 4 presents the assessment of questionable and outlying performance for different parameters. The parameters evaluated include specific gravity, fineness, compressive strength 7-day, and soundness by autoclave.

For specific gravity, there were 7 observations considered in the analysis. The data shows that there were no instances of questionable performance (with z-scores between 2 and 3) or outlying performance (with z-scores greater than or equal to 3). This implies that all the specific gravity measurements fell within an acceptable range without any significant deviations.

Similarly, for the fineness parameter, there were 6 observations considered. No questionable performance or outlying performance was observed, indicating the absence of measurements that deviated substantially from the expected values within this dataset.

Moving on to the compressive strength at 7-day duration, the analysis was based on 7 observations. As with the previous parameters, no questionable or outlying performances were identified, indicating that the compressive strength measurements were within the expected range without any significant anomalies.

Lastly, for soundness by autoclave, 6 observations were considered. Similar to the previous cases, there were no instances of questionable performance or outlying performance. This suggests that the soundness by autoclave measurements exhibited consistency and conformity within the analysed dataset.

In summary, the data provided in Table 4 demonstrates that across different parameters (specific gravity, fineness, compressive strength at 7-day duration, and soundness by autoclave), no questionable or outlying performances were detected. This indicates that the measurements were generally reliable, without any extreme values or notable deviations that could raise concerns about the data's validity or accuracy.

Table 5*. Results of Proficiency Testing.*

Participant Lab Code	Specific Gravity g/cc		Fineness m^2/kg		Compressive Strength-7d %		Soundness by Autoclave %	
	Lab Result	Z' Score	Lab Result	Z' Score	Lab Result	Z' Score	Lab Result	Z' Score
A	2.66	-1.0	404	-0.4	88	-0.4	0.00	-0.9
B	2.70	0.1	394	-1.0	92	1.1	-	-
C	2.75	1.4	402	-0.5	91	0.7	0.04	1.5
D	2.68	-0.4	419	0.4	86	-1.1	0.01	-0.3
E	2.70	0.1	433	1.2	89	0.0	0.00	-0.9
F	2.67	-0.7	418	0.3	87	-0.7	0.01	-0.3
G	2.72	0.6	-	-	90	0.4	0.02	0.3

7. Results and Discussion

The results of the proficiency testing for the fly ash sample provide valuable insights into the performance of different participant laboratories. The specific gravity measurements exhibited some variation among the labs, with results ranging from 2.66 g/cc to 2.75 g/cc. Labs B, D, E, F, and G obtained results within a relatively narrow range, with minor deviations from the assigned value. However, Labs A and C had results that deviated further from the expected value, with Lab A obtaining a lower specific gravity measurement and Lab C obtaining a higher measurement. These deviations are reflected in the corresponding z-scores, which indicate the degree of deviation from the expected value. It is noteworthy that the deviation for Lab C was relatively large, with a z-score of 1.4, highlighting a significant difference from the assigned value.

Regarding the fineness parameter, Labs A, B, C, D, E, and F reported measurements falling within a range of 394 m2/kg to 433 m2/kg. Labs B and D had results that deviated more significantly from the expected value, with z-scores of -1.0 and 0.4, respectively. The other labs demonstrated relatively closer agreement with the assigned value. This suggests that Labs B and D may need to review their methods or techniques to minimize the variability observed in their fineness measurements.

Analysing the compressive strength at 7 days, Labs B and C achieved results that deviated from the assigned value, with z-scores of 1.1 and 0.7, respectively. These labs reported higher compressive strength measurements compared to the other participants. On the other hand, Labs A, D, E, and F had results that were closer to the expected value. It is crucial for Labs B and C to evaluate their testing methodologies or procedures to identify factors contributing to the observed deviations and work towards enhancing the accuracy and reliability of their results.

The soundness by autoclave measurements indicated good agreement among the participant labs, as all the reported values fell within a narrow range from 0.00% to 0.04%. The majority of labs exhibited results with z-scores close to zero, indicating a minimal deviation. However, Lab C reported a higher soundness value, resulting in a z-score of 1.5, highlighting a noticeable deviation from the assigned value. Lab A, on the other hand, reported a lower soundness measurement, resulting in a z-score of -0.9.

Table 5 presents the results of the proficiency testing along with the Z' Scores for each parameter and participant laboratory. The data from Table 5 can be correlated with the bar chart (Figure 1) by matching the Z' Scores with the corresponding bars representing each laboratory's performance. The Z' Score is a standardized score that indicates how many standard deviations a lab's result is from the assigned value. A Z' Score within ±2.0 is considered satisfactory, while scores beyond ±3.0 are considered outliers, and scores between ±2.0 and ±3.0 are questionable.

In conclusion, the proficiency testing results highlight variations in the performance of different participant labs across the specific gravity, fineness, compressive strength at 7 days, and soundness by autoclave parameters. The deviations observed in specific gravity and soundness by autoclave measurements, particularly by Labs A and C, require careful examination and potential improvement to ensure better conformity with the expected values. For Labs B and D, it is important to evaluate their testing methodologies for fineness and compressive strength, respectively, to address the observed deviations and enhance the accuracy and reliability of their results. These findings underscore the significance of proficiency testing for maintaining the quality and consistency of laboratory measurements in the field of material characterization and analysis.

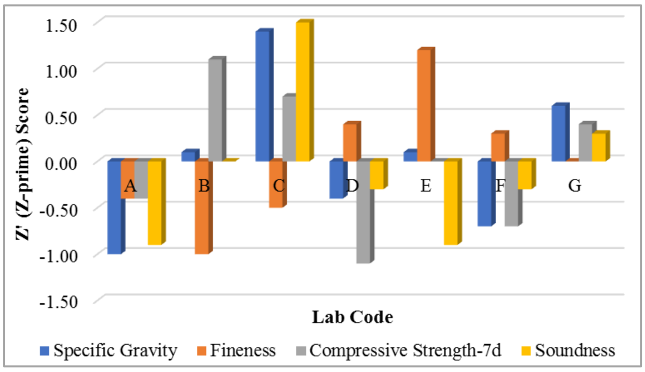

Figure 1. *Bar Chart for Z' (Z-prime) Scores.*

8. Conclusion

Proficiency testing programs (PTPs) demonstrate their critical role in safeguarding the quality and consistency of fly ash testing. This analysis of a specific PTP reveals valuable insights into the performance of participating laboratories across key parameters: specific gravity, fineness, compressive strength, and soundness by autoclave.

While most laboratories exhibited commendable adherence to expected values, some deviations raise concerns and highlight the need for continuous improvement. Labs A and C, for instance, showed discrepancies in specific gravity and soundness by autoclave, prompting the need for a closer examination of their testing methodologies.

Furthermore, Labs B and D faced challenges with fineness and compressive strength measurements, respectively. Evaluating their procedures and identifying factors contributing to these deviations are crucial steps towards enhanced accuracy and reliability.

The PTP's success underlines the significance of such programs in ensuring responsible and sustainable fly ash utilization. Moving forward, continued development and refinement of PTPs, focusing on emerging applications and potential challenges, are essential. By actively participating in and learning from PTPs, laboratories can not only strengthen their competence but also contribute significantly to a more sustainable future for the fly ash industry.

Abbreviations

PT: Proficiency Testing
PTP: Proficiency Testing Program
SD: Standard Deviation
SDPA: Predetermined Standard Deviation
Z' Score: Z-prime Score

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Conflicts of Interest

The authors declare no conflicts of interest.

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