

On the Empirical Study of Elemental Analysis and Metal Testing using XRF Spectrum Analysis Algorithm

Girish Balasubramanian, Senthil Arumugam Muthukumaraswamy

Abstract— This paper proposes an algorithm to perform quick X-ray Fluorescence (XRF) spectrum analysis to be used for metal testing and quality control by empirical studies. The metal industry usually deals with large volumes of scrap and metal during recycling. High speed sorting and testing of these metals allow for increased workflow while ensuring accurate identification of these metals. In this paper, An XRF spectrometer (X-MET8000 by Oxford Instruments) was used to gather raw spectra from some alloys. These spectra were fed into a MATLAB script that was designed to implement an algorithm which quickly identifies the elemental content of the sample by comparing it with the spectra input using Pearson correlation coefficients. Spectra from different alloys were analysed using the proposed XRF analyser algorithm in order to accelerate the identification of the elemental content of the sample. The proposed algorithm is successfully proved for the empirical method to which effectively identifies the elemental composition of the sample. This algorithm can also be used for testing of hazardous material. The elemental composition is then compared with the standard benchmark samples of the required alloy for verification as a measure of quality control and a pass is given for matching the composition of the alloy. The computational efficiency of the proposed algorithm is also tested using the MATLAB profiler and was observed to be around 0.003 seconds for quality control and 0.056 seconds for alloy identification.

Index Terms— X-ray fluorescence, Elemental analysis, Spectrum Analysis, Metal industries, Quality Control, Pearson correlation coefficient.

I. INTRODUCTION

Metal and scrap industries require accurate metal identification techniques to ensure quality. Alloys may be purchased incorrectly or can be contaminated with hazardous or low quality substances that degrade the quality of the metal. Fast metal identification techniques can save the industry time and money by providing verification of alloys before the industry makes a purchase [6]. One of the commonly used practices for this purpose is known as X-ray Fluorescence (XRF) spectrum analysis. Each sample subject to XRF provides a unique spectrum that can be analyzed to identify the elemental content present in that sample

Currently, in metal industries, this XRF spectrum analysis is generally done via handheld XRF instruments which consist of an X-ray tube, a detector and a processor to perform analysis. This can prove to be a potential safety

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concern depending on the user as X-rays are emitted from the device. However, XRF spectrum analyzers are nondestructive as compared to the other types of spectrum analyzers such as laser induced breakdown spectroscopy (LIBS) or optical emission spectroscopy (OES). A computer aided approach provides a safer route to perform the same action and hence a MATLAB program is used to implement this proposed algorithm.

This paper proposes an algorithm that performs analysis of XRF spectra to identify the element composition of the spectra and hence the alloy. The algorithm can be implemented in a separate computer to take in spectra obtained from any X-ray spectrometer or can also be used by XRF analyzers. The algorithm was designed to perform two functions: it could either check whether the spectra belonged to the specific meta/alloy (quality check: pass/fail) or identify the alloy corresponded to that spectra (identification). Both of these qualitative methods were tested empirically using the proposed algorithm and their efficacy is successfully demonstrated.

A MATLAB script was then devised for the proposed algorithm in order to identify the elemental content of XRF spectra based on the XRF characteristics of each element and tested by passing known alloy spectra into it. Then the identification and testing are validated by comparing the results obtained through MATLAB simulation results.

In Section II, the different XRF techniques those used for elemental analysis are discussed. The technique proposed in this paper describes the details of Pearson correlation coefficients (PCC) and their influence on the empirical studies of elemental analysis and metal testing processes. The XRF spectra used for testing were obtained using an XRF instrument (X-MET8000 by Oxford Instruments) [17] and a database was created containing spectra of various alloys. The features of the XRF instrument and how the instrument was used to obtain test spectra for the algorithm are also showcased in section II. It also discusses more in detail about the current analysis techniques employed by the metal industries [18].

In Section III, the working of the proposed algorithm is discussed and also describes how this algorithm is tested using MATLAB simulation. In Section IV, the MATLAB simulation results for the metal testing (quality control) and element analysis (identification) are analyzed. The computational efficiency (execution time) of the proposed algorithm is also tested using the MATLAB profiler. Based on the results obtained and discussions, proper conclusion are drawn and stated in section V. The scope for the future work also mentioned.

II. BACKGROUND STUDY

2.1 XRF Techniques

X-Ray Fluorescence is an analytical technique widely used to determine the elemental composition of different types of materials. It works by emitting a primary x-ray from a source onto a sample. The atoms of the sample then emit a fluorescent x-ray that is measured usually by a detector. Depending on the element, x-ray at either K or L level energies are emitted [7]. The intensity of x-ray at these energies can be related to elements to identify the type of a particular element in a given sample of metal and/or alloys.

In some applications or industries, qualitative analysis provides sufficient information to speed up the process. These include cases where sorting between various pure metals and impure metals is required, irrespective of the composition. In these cases, no extra calibrations methods are needed and no specific conditions are to be met. However, there are also some cases where the composition is also important.

In order to quantitatively analyze the content, two types of methods can be applied. These include,

1. Fundamental Parameter (FP) – Relationship between the measured X-Ray intensities and concentrations of elements in the sample.
2. Empirical – Relating the existing and known standard spectra with measured spectra.

The MATLAB script is designed to implement the XRF analyzer algorithm using the empirical method for qualitative analysis. In order to apply the Empirical method, four conditions are required to be considered [16]. These include,

1. The sample must not contain layers or rusting etc.
2. Have a flat sample
3. The sample must meet the infinite thickness condition (no x-rays escape to the other side of the sample).

This is to ensure that the spectra obtained is pure and is not affected by external factors.

2.2 Pearson correlation coefficient

The Pearson correlation coefficients (PCC) are used as a measure of linear correlation between two variables [2]. It can be used to how closely two variables are related. The PCC between two random variables a and b is defined as given in (1),

$$\rho(a, b) = \frac{E(ab)}{\sigma_a \sigma_b} \quad (1)$$

Where, $E(ab)$ is the cross-correlation between a and b . σ_a and σ_b represent the standard deviation of the a and b respectively.

In the case of XRF spectra, the PCCs are used to identify the similarity between two spectra. Hence the equation (1)

needs to be slightly modified. Let A be the vector for input spectrum data and B be the vector for spectrum data of the alloy to be compared [1-4] and these vectors are defined as shown in (2),

$$A = [a_1 a_2 \dots a_L]^T, B = [b_1 b_2 \dots b_L]^T \quad (2)$$

The Pearson correlation coefficients between A and B can be found by using (3),

$$\rho(A, B) = \frac{E(A^T B)}{\sqrt{E(A^T A)E(B^T B)}} \quad (3)$$

Where $E(A^T B)$ is the cross-correlation between A transposed and B vectors and $-1 \leq \rho^2(A, B) \leq 1$.

This calculation will yield a result between -1 and 1. If the PCC value is close to 1 then the two spectra are similar. Negative values indicate a negative correlation while 0 indicates no correlation. The similarity limits can be set as desired depending on the desired accuracy level. Although, $\rho(A, B) = 1$ represents an exact match, it is not practically possible to attain this value. Hence, a typical value of 0.95 was chosen as the threshold point to decide on the suitable match for pass/fail operation. In identification operation, the highest value will be chosen. Another advantage of PCC is that it eliminates time taken per XRF sample as a factor during analysis.

2.3 XRF Instrument

The XRF instrument, X-MET8000 by Oxford Instruments [17] is used to obtain spectra for different metals. This instrument uses a high performance X-ray tube and a silicon drift detector (SDD) to identify the spectra of various samples. The instrument can detect energy levels from 0 – 51.33 keV in 25.063 eV increments. Based on these information, the total number of data points present in each spectrum is around 2048. The spectra obtained from the instrument are plotted in counts vs. energy levels (keV). Fig.1. shows the spectrum of silver obtained using the XRF instrument, X-MET8000.

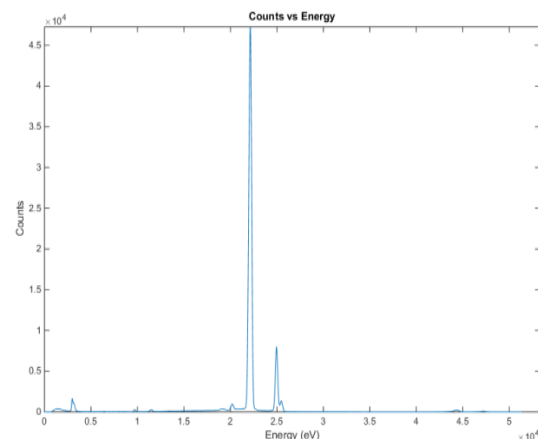


Fig. 1. Spectrum of silver obtained using X-MET8000

Although each element has a characteristic energy level pair, the counts can vary depending on the duration that the element was exposed to the x-ray. For the purpose of

obtained test spectra, each sample was subject to 15 seconds for each test to obtain uniform results.

2.4 Current industrial analysis techniques

Current industries use many different types of analytical techniques ranging from handheld XRF, bench top XRF, LIBS and OES. Each analytical technique comes with their pros and cons [18]. The primary objective of each of these techniques is to qualitatively or quantitatively analyze elements presented in the metal/alloy.

Both OES and LIBS are atomic emission techniques. The LIBS method creates plasma which excites the elements and OES uses either an arc or spark between an electrode and the sample. Since OES requires a conductive sample to generate this arc or spark. It is restricted to metal analysis. For this application of metal testing, this is not a problem. LIBS is virtually nondestructive. It uses 1 nanogram of a sample per test. This is up to the industries to decide on how much is an inconvenience as it leaves a mark on the sample. However, OES removes far more material (1 microgram) and hence considered destructive.

As XRF uses a radiation source as described earlier in section 2.1, it has advantages as compared to the other two analysis methods (LIBS and OES) in that it is completely nondestructive (no sample used). Another advantage of XRF analysis is that the XRF spectra contain minimal number of characteristic lines as compared to LIBS or OES. Fig.2 show how a LIBS spectrum looks like.

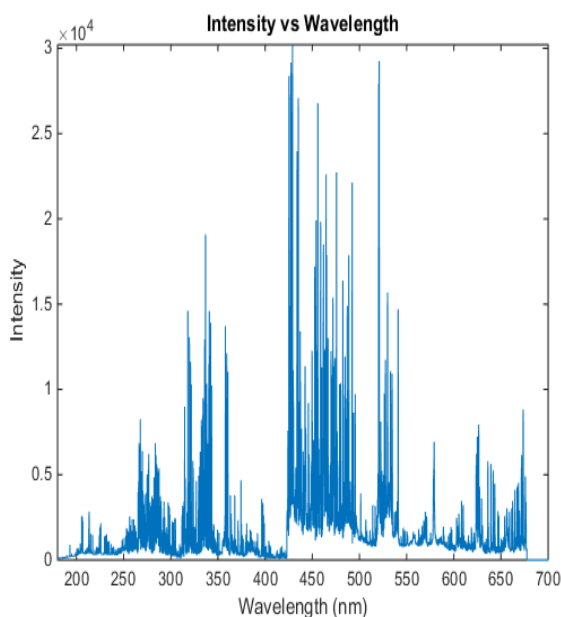


Fig. 2 LIBS spectra for chromium

However, there is a significant drawback in XRF as it uses radiation. Due to this, some industries may require health and safety control certification or a license to handle such radiation emitting equipment. OES and LIBS on the other hand do not require this since these methods do not emit radiation. Another notable disadvantage of XRF is its inability to detect carbon.

III. ALGORITHM AND METHOD

The proposed algorithm is implemented using MATLAB initially adopts two stages for both quality control (pass/fail operation) testing and identification operation:

1. Use peak identification to find peaks present in the spectral content of the sample.
2. Relate the peaks found to elements using known characteristics.

Then for the quality testing on deciding the pass/fail operation. Three more steps are carried out:

3. Compare the spectrum input with the spectrum of the required alloy using Pearson correlation coefficients [1-4].
4. Check whether the coefficients are at least 0.95.
5. If the condition is met, the alloy matches the input spectrum and it is a pass, else it is a fail

Otherwise, the following two steps are carried out for identification operation:

3. Compare the spectrum input with the database of alloys using Pearson correlation coefficients. Compare only with the alloys that contain the elements that were identified.
4. Identify the alloy that provided the best match (highest coefficient) with reference to a threshold value, of say 0.95 out of 1.

2.1 Peak identification and element characteristics

The peaks present in an XRF spectrum is identified by using the *peakfind* function present in MATLAB. This function was set to identify all peaks higher than a threshold of 1000 counts in the spectra. The element can be determined by relating the peaks to the characteristic energy level of that particular element. Some of the elements present in alloys commonly used in metal and scrap industries and their respective characteristic energy levels are shown in Table 1 [7].

Table 1: Elements commonly used in metal industries and their characteristic XRF energy level

Element	K - series		L - series	
	K α (keV)	K β (keV)	L α (keV)	L β (keV)
Al	1.487	1.557	-	-
Ag	22.163	26.095	-	-
Au	-	-	9.705	11.609
Cr	5.415	5.947	-	-
Cu	8.048	8.905	-	-
Fe	6.404	7.058	-	-
Mn	5.899	6.490	-	-
Ni	8.265	7.478	-	-
Pb	-	-	10.555	12.791
Pt	-	-	9.439	11.231
Sn	25.044	28.439	-	-
Ti	4.511	4.932	-	-
W	-	-	8.396	9.819
Zn	8.639	9.572	-	-
Zr	15.770	17.662	-	-

2.3 Alloy database construction

The database consisting of alloys are constructed by obtaining the XRF spectra of known alloy standards and storing the spectra data in MATLAB variables. Some of the few alloys listed in the database [5] including their known composition are tabulated in Table 2.

Table 2 : Some of the alloys present in database

Alloy	Known Composition
CDA836	85% Cu, 5% Pb, 5% Sn, 5% Zn
CDA857	60.9% Cu, 1.58% Pb, 36.1% Zn
CDA863	63% Cu, 6.2% Al, 3% Fe, 3.7% Mn, 25% Zn
CDA936	81% Cu, 12% Pb, 7% Sn
CDA964	66.7% Cu, 30% Ni, 1% Mn
NIT 60	17% Cr, 8% Mn, 8.5% Ni, 4% Si, Base Fe
C89325	86% Cu, 3% Bi, 10% Sn, 1% Zn
C83450	88% Cu, 2% Pb, 2.5% Sn, 6.5% Zn, 1.6% Ni
317	19% Cr, 13% Ni, 3.5% Mo, Base Fe
356	90% Al, 9% Si

IV. RESULTS AND DISCUSSION

The MATLAB simulation results for the proposed XRF analyzer algorithm for both quality control testing (pass/fail operation) and the qualitative elemental analyses (identification) are presented for a few selected samples of alloys, CDA386, CDA857, CDA863 and NIT60, from the database. Profiling is also performed on the MATLAB code for the same samples and the results are shown in this section.

3.1 Pass/Fail operation

In order to test the Pass/Fail operation, the script was set to check the spectra for a known alloy. In this case, CDA836 was used as the testing alloy. The script was checked for both pass and fail by inputting a correct spectra and inputting a wrong spectra. Fig. 3 shows the spectrum of the required alloy CDA 836.

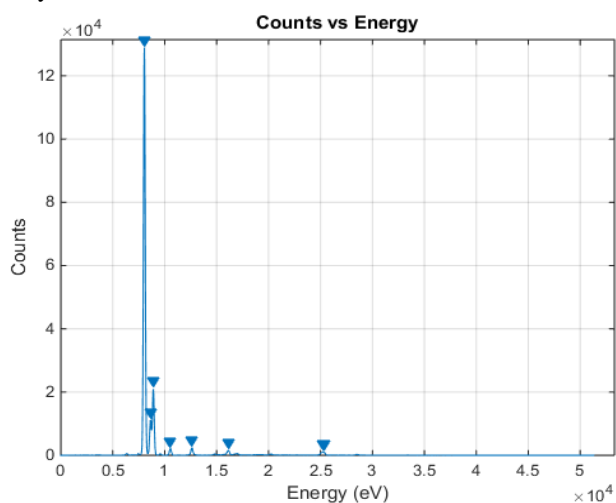


Fig. 3. Spectrum of alloy CDA836 obtained through the XRF instrument

To test the pass condition, another spectrum of the same sample alloy CDA836 was used. Fig. 4 shows the spectrum of the input alloy.

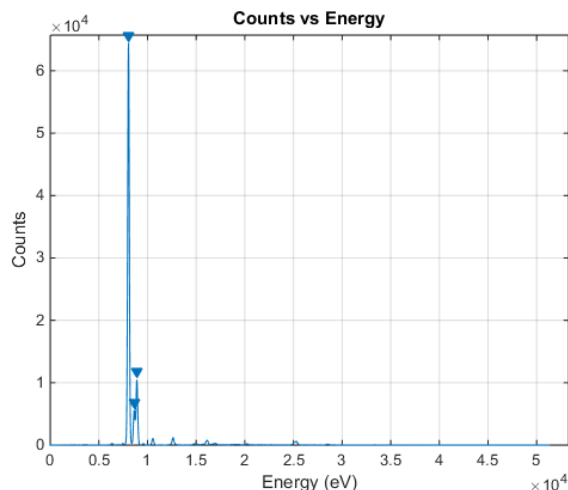


Fig. 4. Spectrum of the input used for pass check (CDA836)

It was observed by sight that the input spectrum did not match exactly with the required spectrum. This is because the XRF spectrum may be obtained differently. Using the PCC values, the identification of the alloy can be verified. Fig. 5 shows the elements present within the input spectrum that was obtained from the MATLAB script.

```

-----Starting XRF Test-----
-----Testing Spectra Qualitative-----
Copper - Yes
Lead - Yes
Tin - Yes
Zinc - Yes
    
```

Fig. 5. Elements identification result by means of MATLAB script

It was observed that the elements identified matched those present in alloy CDA836. However, the compositions may vary. Therefore this result alone might not be enough to provide justification to pass the quality control check. The PCC was then calculated between both the input and the known spectrum of CDA836. The result obtained through the script is shown in Fig. 6.

```

similarity =

    1.0000    0.9890
    0.9890    1.0000
    
```

Fig. 6. Similarity check results for pass check

The similarity coefficient was found to be 0.989. This value is higher than the threshold value of 0.95. Hence, it can be confirmed that the input spectrum correctly identifies the alloy CDA836. This means it is a pass on the quality check.

In order to test a fail, the input spectrum was changed to be the spectrum of CDA857. The XRF spectrum of CDA857 that was used as the test input is shown in Fig. 7.

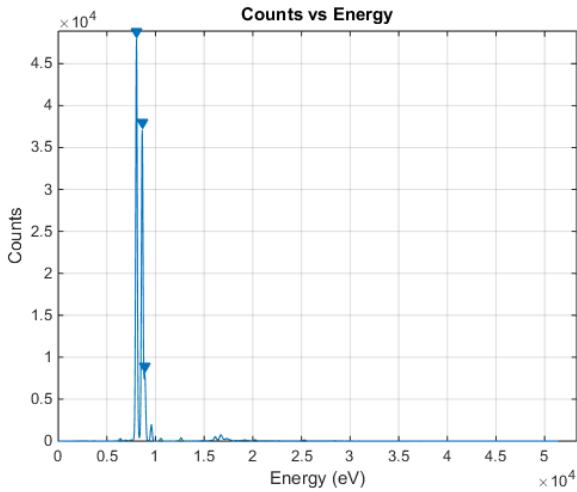


Fig. 7. XRF spectrum of CDA857 obtained through the XRF instrument

First, the element identification was performed. The result of element identification is shown in Fig. 8.

```

-----Starting XRF Test-----
-----Testing Spectra Qualitative-----
Copper - Yes
Lead - Yes
Zinc - Yes
    
```

Fig. 8. Element identification results for CDA857

Although the elements were correctly identified for CDA857, the required elements of CDA836 were not identified. The test may be stopped here with a fail. However, the PCC was calculated for the purpose of demonstration. The results of performing the PCC are shown in Fig. 9.

similarity =	
1.0000	0.8397
0.8397	1.0000

Fig. 9. Similarity check results for a fail check

The similarity coefficient for this case is 0.8397 which is less than the threshold value, 0.95. Hence, it can be said that input spectrum did not match with the required alloy CDA836.

Performing the pass/fail check for both conditions proved to be successful as the algorithm implemented using the MATLAB script correctly identified both scenarios during when there is a pass and when there is a fail. Similarly, any alloy can be compared with another using this algorithm as long as the required alloy is present in or added to the database.

3.2 Alloy identification operation

The alloy identification operation identifies the closest alloy present within the input spectrum. In order to test the alloy identification operation, three alloys were tested. In this case, alloy NIT 60, CDA857 and CDA863 were tested using this operation. The XRF spectrum of NIT 60 and CDA863 are shown in Fig. 10 and Fig. 11 respectively.

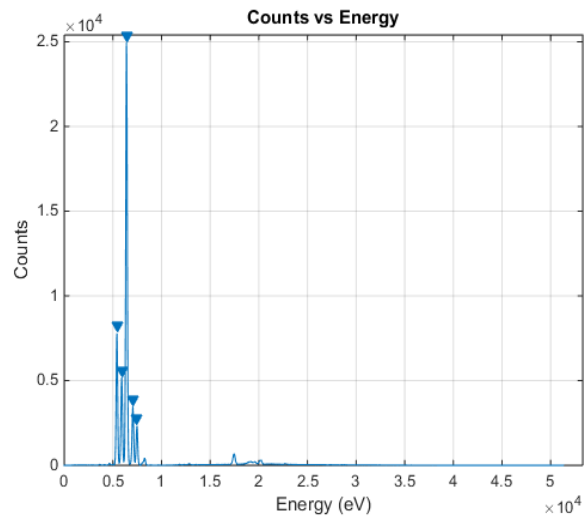


Fig. 10. XRF spectrum of NIT 60 obtained through the XRF instrument

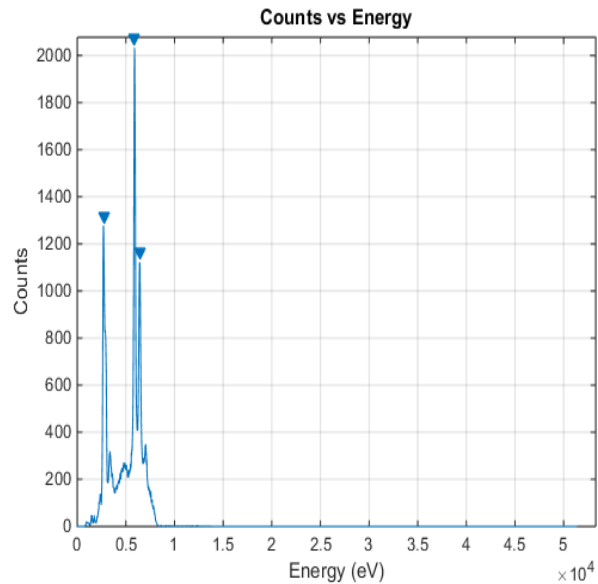


Fig. 11. CDA863 spectra obtained through the XRF instrument

The element identification results for NIT 60 are shown in Fig. 12 and it was observed that the elements were correctly identified.

```

-----Starting XRF Test-----
-----Testing Spectra Qualitative-----
Chromium - Yes
Iron - Yes
Manganese - Yes
Nickel - Yes
Silicon - Yes
    
```

Fig. 12. Element identification results for NIT 60

The elements were identified correctly. The alloy identification operation identifies the highest similarity from within the database using PCC and matches it to the alloy. The alloy identification results are shown in Fig. 13.

```
-----Alloy Identification Results-----
NIT 60, PCC = 1.000, Perfect Match
CDA857, PCC = -0.0050, No Match
CDA836, PCC = -0.0013, No Match
```

Fig. 13. Alloy ID results for NIT60

It was observed that the MATLAB script identified the spectrum exactly while the other two alloys were identified to be no matches. Similarly, the spectrum of CDA857 that was shown in Fig. 5 and the CDA863 alloy were also tested using this method. The alloy identification results are shown in Fig. 14.

```
-----Starting XRF Test-----
-----Alloy Identification Results-----
CDA863 PCC = 1.000, Perfect Match
NIT 60, PCC = 0.5291, No Match
CDA857, PCC = -0.0158, No Match
CDA836, PCC = -0.0080, No Match
```

```
-----Alloy Identification Results-----
CDA857, PCC = 1.000, Perfect Match
NIT 60, PCC = -0.0050, No Match
CDA836, PCC = 0.8397, No Match
```

Fig. 14. Alloy ID results for CDA863 and CDA857

From the results presented in Fig. 13 and Fig. 14, it was observed that the proposed algorithm which is implemented with MATLAB script appropriately identified the alloys that were tested.

3.3 Profiling

The computational efficiency of the proposed algorithm is tested using the MATLAB profiler [19]. The profiler identifies the execution time of each parts of the code while showing the other processes that were carried out. This helps by eliminating any time that was taken by human interaction to purely identify the time taken by the processing of the code itself. The profiler was accessed through the MATLAB window by using the “profile viewer” command input to the

command window. The window used for profiling is shown in Fig. 15.

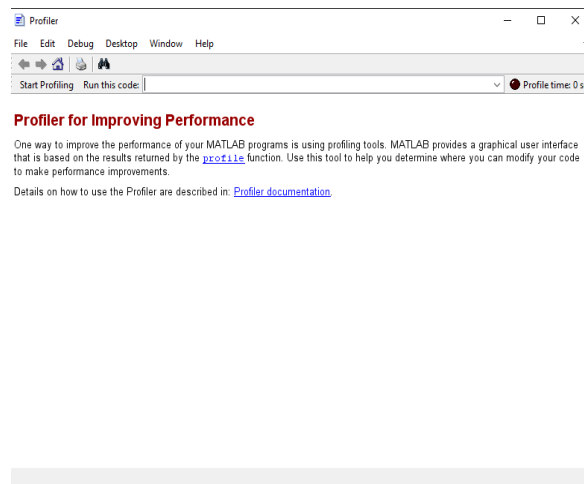


Fig. 15 Profiling window used to measure execution time in MATLAB.

Both operations were tested using the profiler to run for the same alloys as before measuring the execution time. The results obtained for the time executed were presented in Table 3.

From the profiling window, the time taken for processing the pass/fail operation is 0.003 seconds for processing excluding the time taken to load the data as this can depend on user interaction. Similarly, the alloy identification execution time was measured as 0.056 seconds on average. It was expected that the alloy identification would take more time compared to the pass/fail operation as it is also directly proportional to the amount of spectra present within the database. Depending on the alloys present within the database, the execution time for alloy identification may increase. This can be optimized by creating separate databases for each work environment only consisting of the most relevant alloys required for the application.

Table 3: Summary of results obtained along with execution times

Mode	Alloy	Test Alloy	Expected Result	Actual Result	Successful	Execution Time (s)
Pass/Fail	CDA863	CDA836	Fail	Fail	Yes	0.0028
	CDA836	CDA836	Pass	Pass	Yes	0.003
	CDA857	CDA857	Pass	Pass	Yes	0.003
	NIT60	CDA836	Fail	Fail	Yes	0.0029
	Average Time					
Alloy Identification	CDA863	-	CDA863	CDA863	Yes	0.062
	CDA836	-	CDA836	CDA836	Yes	0.052
	CDA857	-	CDA857	CDA857	Yes	0.053
	NIT60	-	NIT60	NIT60	Yes	0.056
	Average Time					

V. CONCLUSION

From the obtained results and tests, the MATLAB script used was able to execute the proposed XRF analyzer algorithm accurately with reliable results. It is also observed the efficacy of the proposed algorithm used for both quality control check and alloy identification. The proposed elemental analysis and the empirical studies can greatly improve the efficiency of metal or alloy testing and quality control in metal industries. The proposed XRF analyzer algorithm may be further extended into many other applications where identification of XRF spectra is required.

The pass/fail operation correctly identified a spectra of CDA836 obtained through the same instrument but different timing setting and also correctly identified a fail when another alloy was used as an input. Moreover, the alloy identification operation was able to correctly identify both alloys CDA857 as well as NIT60 by comparing the similarities of their spectra to the ones stored in the database via PCC. Moreover, the computational speed of the pass/fail process for the proposed algorithm is 0.003 seconds per sample. This is a very good measure on the computational efficacy of the proposed algorithm and able to handle batch identification at a rapid rate. Although, the processing time for alloy identification 0.056 seconds, this result may vary depending on the size of the database, it is still a faster process for industrial purposes.

Using a computer aided approach improves the efficiency as well as safety. Since, using hardware XRF instruments is potentially dangerous with continuous exposure and thus allowing machines to perform such tasks using computer algorithm is a safer approach for the quality control and testing aspect of a metal industry.

ACKNOWLEDGMENT

Thanks to Hillsborough General Trading LLC. Dubai for providing the equipment used to perform this study and a special thanks to Oxford Instruments U.K for manufacturing a powerful tool capable of obtaining XRF spectra fast, efficient and accurately.

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