Optimizing Copper Production from Scrap: The Impact of Heat Treatments on Purity and Yield

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Abstract: R Recent research has focused on improving the copper purification process to meet the strict purity requirements, particularly in sectors such as aerospace. This study investigates the use of thermal refinings on samples of copper scrap before melting at specific temperatures between 373 K and 1223 K. These samples were melted and then cast at 1393 K, which has been confirmed in previous studies as the optimum temperature to achieve higher purity from copper scrap. The first thermal refinements consist of continuous heating with isothermal holds at the specified temperatures, with each refining having a holding time of 5, 10, 15, or 20 minutes set at the beginning. By exploiting the volatility of certain impurities (Zn, As and P) and the oxidation of others, this process has significantly improved the purity of the casting copper from 99.9230 .wt% to 99.9714 .wt%. Despite these improvements, significant mass losses were observed at the end of thermal refining for equivalent holding times greater than 10 minutes. These mass losses are mainly due to degradation of thin copper wire. To remedy this, thermal refinings with different holding times were tested. One of these latest refinings not only improved the purity to 99.9716.wt% and reached a thermal conductivity of approximately 360 W/m·K , but also reduced the total refining holding time to 94 min. These results help optimize copper purification processes by reducing costs and mass losses while improving overall efficiency.

Keywords: Thermal refining; Purity; Isothermal holds; Holding times; Volatility of impurities; mass losses.

1. Introduction

The need for effective resource management has become increasingly critical, particularly with the emergence of the circular economy, which promotes a more judicious use of materials. This approach emphasizes material reuse and recycling to reduce waste and preserve our valuable natural resources [1-2-3]. A concrete example of this approach is copper recycling, a metal widely used in numerous industries. Despite significant growth in copper consumption and primary production in recent years, secondary production has remained stagnant [4]. This situation is particularly notable given that the average lifespan of a tonne of copper in use is 23 years, meaning that a tonne of copper reaching the end of its life today would have been processed and manufactured around the turn of the millennium [5]. This stagnation highlights the challenge facing the copper industry in integrating more sustainable practices while considering issues related to energy efficiency, greenhouse gas emissions reduction, and climate change mitigation.

Recycled scrap [6] used to produce secondary copper can be sourced from old metal scrap and end-of-life products, as well as new scrap generated during the production of copper-containing goods. New scrap is inexpensive to recycle because it is easy to identify, sort, concentrate, and transport [4]. However, old stored scrap that has accumulated in increasing quantities over the past 40 years must be discerned and separated by direct [7-8-9-10] or indirect [11] methods before recycling. Generally, waste is processed to remove high melting point impurities such as ceramics as this increases the recycling energy consumption. Companies that only have melting furnaces without refining equipment are limited to using high-purity waste, such as industrial waste (usually provided by their own customers), No. 1 copper and wire scrap. In contrast, those that incorporate a refining step into their process can also handle enameled and wound wires, transformer windings, cut radiators and No. 2 copper. As for foundries, they accept a wide variety of materials, even those containing low quantities of copper [11].

Most of the research in copper purification has utilized either anode residues after an electrolysis process [12], anode refining furnaces[13], an electrolytic copper plate [14], an electrolysis product [15], electronic waste [16-17], molten copper scrap [18-19], polymer-coated copper wires (WCW) [20], blister copper [21-22], copper bars [23-24] , or copper concentrate [25]. By conducting experiments under specific conditions, such as in a vacuum (with or without expansion) or using chemical agents, these research efforts have achieved the production of 5N copper (99.999 .wt%) through pyrometallurgy and 6N copper (99.9999 .wt%) through hydrometallurgy. In theory, thermal refining exploits the volatilization of certain impurities, whose pressures are higher than that of copper, in the following order: Rb > Te > Cd > Mg > Sr > Li > Sb > Ca > Bi > Pb > Al > Pd > In > Ag > Th > Sn > Cu > Ge > Cr > Co > Ti > U > V, thereby ensuring an improvement in the purity of the cast copper.

In recent years, China « the largest transformer of copper waste » has decreased its imports of both low-quality copper scrap (category 7) and higher-quality copper scrap (category 6) as part of its 'green' policy .This reduction has led to the development of asmall industry in many countries, notably Morocco, which produces approximately 22,000 tons of copper scrap. In order to enable this industry to produce copper with a purity close to 4N under ambient conditions, this article will focus on experimenting with a thermal pre-treatment process in an industrial environment – a plant without refining equipment – on copper scrap to produce semi-finished copper with the highest possible purity. This thermal pre-treatment process has reduced impurity level and improved the purity of the copper obtained by melting the scrap at 1393 K, resulting in a higher purity level than that achieved in the previous study [15-26].

2. Methodology

The experiments were conducted at the Benomar Metal Company (BMC) foundry in Morocco. Samples with an average mass of 498 g (Figure. 1) were selected from the dirtiest copper scrap routinely processed into semi-finished products by this foundry. They are mainly composed of winding wires, radiator tubes, various types of electrical wires, copper plates and ingots, copper scraps, pipework, connecting elements, as well as parts from appliances and automobiles.

Figure. 1: *Type of raw material*

The raw material composing these samples was cleaned with water in a rotary drum to remove surface impurities, especially soil. Thermal refining was conducted in a « Burnout Furnace With Digital Timerbox » electric resistance furnace, which has a power capacity of 3 kWh. This furnace is equipped with a regulator that maintains the required temperatures for the chosen periods. All meltings were conducted at 1393 K in an induction furnace "15KW/HSN-84671110" using a high-density graphite crucible meeting the Singrafine R7510 grade specifications. It is noted that the study [] identified 1393 K as the optimal melting-casting temperature for copper scrap, resulting in higher copper purity.

After melting, the samples were cast as black copper billets (Figure.2). These billets were surfaced and polished before being analysed by mass spectrometry (SPECTROMAXx) to determine their chemical composition. This composition is an average of more than four measurements taken on each billet. The calibration of the measuring instrument was carried out using a standard, guaranteeing an accuracy of 2% [27].

Figure.2 *Products of sample melting*

First, a non-thermally refined sample was melted at 1393 K to determine the approximate chemical composition of its casting. Spectrometric analysis of this casting revealed a copper content of 99.92 .wt%.

For each selected period (5, 10, 15, and 20 min), three tests were conducted. During each test, the sample was continuously heated with isothermal holding stages at temperatures of 473 K, 573 K, 773 K, 973 K, 1123 K, and 1223 K [28]. The duration of each holding stage was equal to the selected period (Figure. 3). After cooling in air, the sample was melted and cast into a graphite mold. The melting process was carried out without using any protective flux.

To determine the efficiency and effectiveness of the pretreatment process, the residues of these pretreatments were analyzed in a private laboratory using X-ray fluorescence (XRF) and plasma optical transmission spectrometry (ICP-OES). These techniques made it possible to precisely determine their chemical composition.

The average thermal conductivity of the sample was determined using the experimental approach described in the previous study [26].

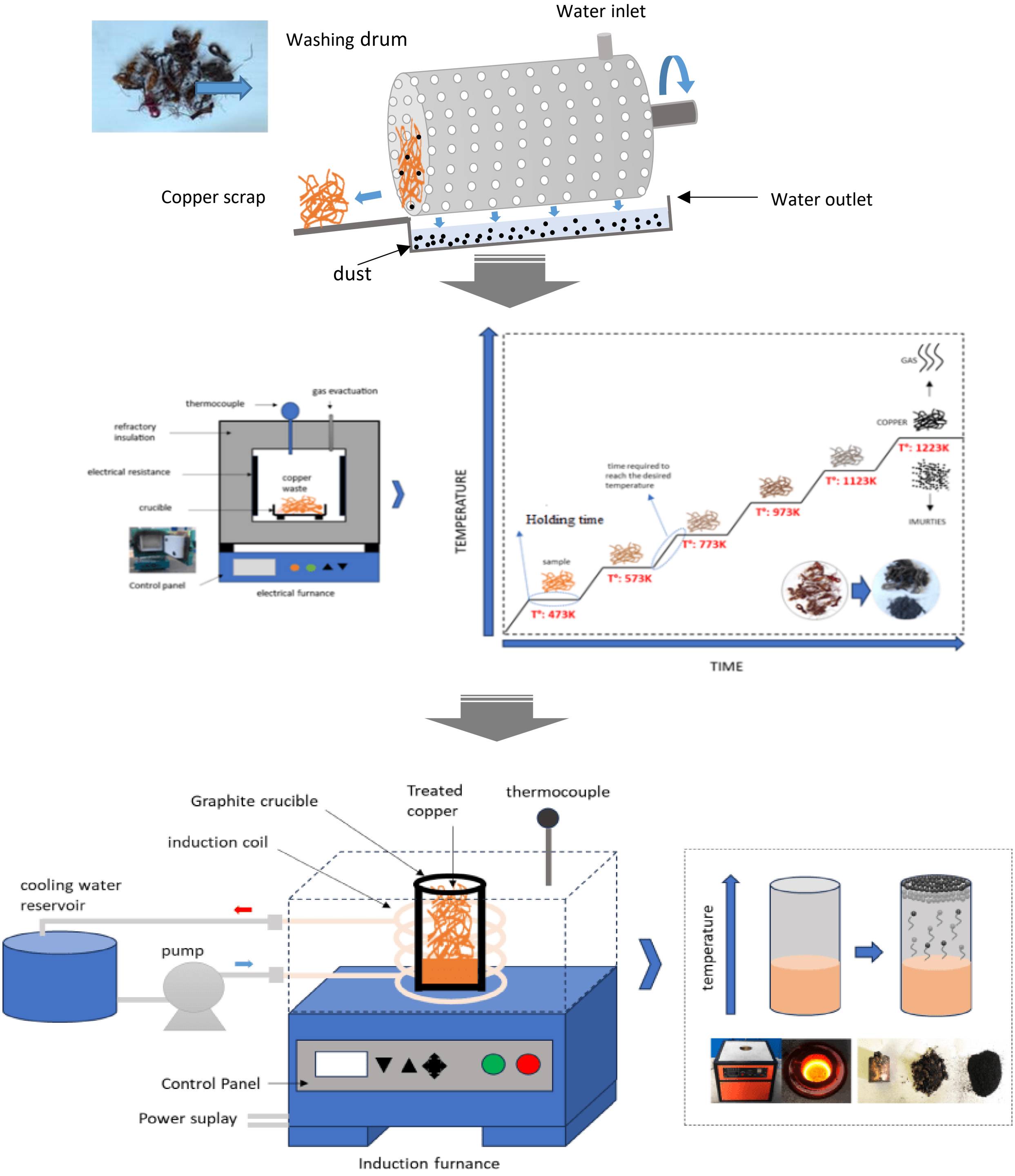


Figure. 3 *Thermal Refining-Melting-Casting Process*

3. Experimentation

3.1 Thermodynamic Study

Figure.4 shows that 15 chemical elements have higher boiling temperatures than copper, while the opposite is observed for the other 16 elements. This last observation will be exploited by the thermal refining process described in this study to reduce the levels of certain impurities (P, Sn, Pb, Zn, Sb, Al and As) whose melting temperatures are lower than its maximum temperature 1223 K. From a thermodynamic point of view, some of these impurities will volatilize during this process according to the data mentioned in table.1 [29-30].

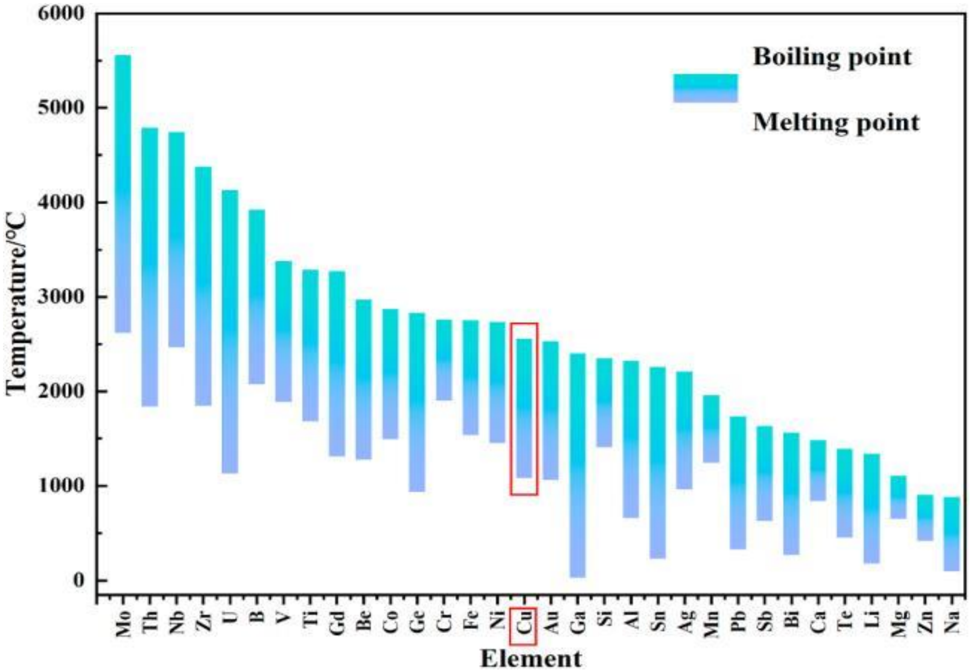


Figure 4. *Diagram of the saturated vapor pressure lgP-T of the impurity element sand copper.*

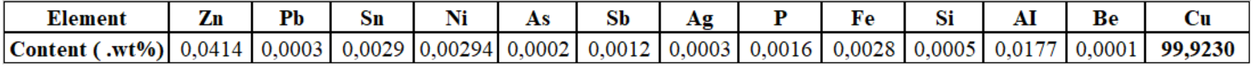
Table 1. *Vapor Pressures of Inorganic Compounds, up to 1 atm*

	Pressure, mmH Temperature, °K										Melting Point °K
	1	5	10	20	40	60	100	200	400	760	
S	456,8	496	516,8	537,7	561,3	578,5	600,2	632,7	672,6	717,6	385,8
Sn*	1765	1907	1976	2050	2128	2176	2241	2336	2442	2543	504,9
Pb*	1246	1372	1435	1507	1582	1631	1694	1792	1903	2017	600,5
Zn	760	831	866	905	946	973	1009	1061	1117	1180	692,4
P	349,6	384,2	401	419,2	439,7	452,8	470,3	295,7	524	553	317,1
As	645	689	710	732	756	771	791	821	852	883	1087
Sb*	1159	1257	1306	1357	1414	1449	1496	1561	1637	1713	903,5
Al*	1557	1694	1760	1828	1908	1957	2022	2117	2220	2329	933
Cu	1901	2068	2152	2243	2340	2400	2480	2598	2738	2868	1356

(*): Elements with melting temperatures above 1223K at atmospheric pressure.

Under ambient conditions, only the Zn, P, and As impurities present in the selected samples will exhibit high vapor pressures during thermal refining (Table 1). Considering the high content of these elements, representing 60% of the total impurities in the cast billet of the untreated sample (Table 2), their reduction will undoubtedly have a significant impact on the successful purification of the copper. It should also be noted that this refining process will facilitate the removal of enamel from the copper wire.

Table 2. *The Copper and Impurities Content in the billet cast from untreated sample*



Element	**Zn**	**Pb**	**Sn**	**Ni**	**As**	**Sb**	**Ag**	**P**	**Fe**	**Si**	**Al**	**Be**	**Cu**
Content (.wt%)	0,0414	0,0003	0,0029	0,00294	0,0002	0,0012	0,0003	0,0016	0,0028	0,0005	0,0177	0,0001	**99,9230**

3.2 Premiers Résultats

Referring to Table 3, it appears evident that the average copper contents of the billets obtained by melting the treated samples varies between 99.9692 and 99.9714 .wt%. This represents an increase of 0.05% compared to that obtained for the billet of untreated sample.

The opposite trend is observed in the sums of impurity values of the treated samples, showing a decrease ranging from 63% to 81% compared to that of the untreated sample. This decline is mainly due to the decrease in the concentrations of volatile impurities (Zn, As, and P) whose sums experience decreases ranging from 0.69% to 0.93% compared to those observed in the untreated sample for these elements (Figure. 5).

Table 3. *Copper and Impurity content in the billets cast from samples*

	Holding Time (mn)	Zn	Pb	Sn	Ni	As	Sb	Ag	P	Fe	Si	AI	Be	Total impurities (.wt%)	Cu
Pre-taitement	**20**	0,00160	0,0003	0,0011	0,00290	0,0002	0,0011	0,0003	0,0011	0,003	0,0005	0,0015	0,0001	**0,01365**	**99,9706**
	15	0,0016	0,0003	0,0009	0,0028	0,0002	0,0011	0,0003	0,0011	0,0031	0,0005	0,0013	0,0001	**0,01334**	**99,9714**
	10	0,0103	0,0003	0,001	0,0032	0,0002	0,00170	0,0003	0,0013	0,003	0,0005	0,0012	0,0001	**0,02304**	**99,9710**
	5	0,0119	0,0003	0,0021	0,003	0,0002	0,0023	0,0003	0,0012	0,0029	0,0005	0,00133	0,0001	**0,02615**	**99,9692**

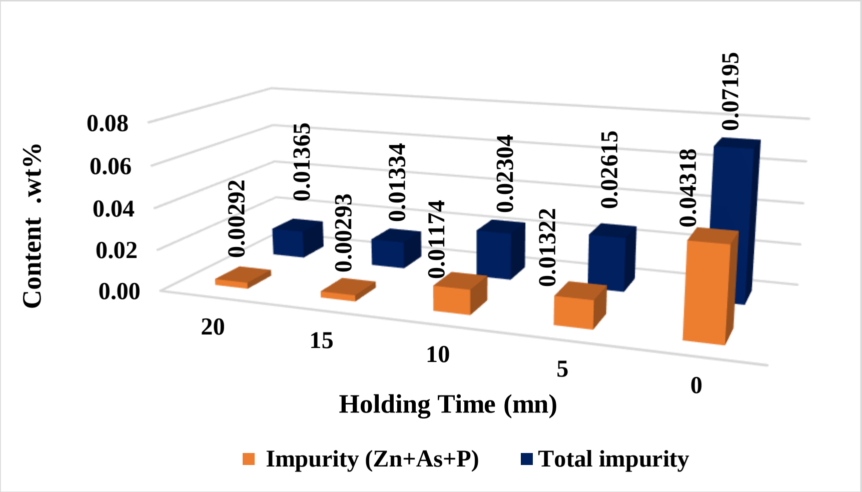


Figure 5. *Variation of impurity content and that of the group (Zn, As, P) in the billets cast from samples with holding time*

It should be noted that the mass losses observed during these thermal refining vary from 1.6% to 2.07%. At the end of the billet casting process, these losses range from 5.7% to 6.21% (Figure 6).These losses, less than 8%, are tolerable at the industrial level.

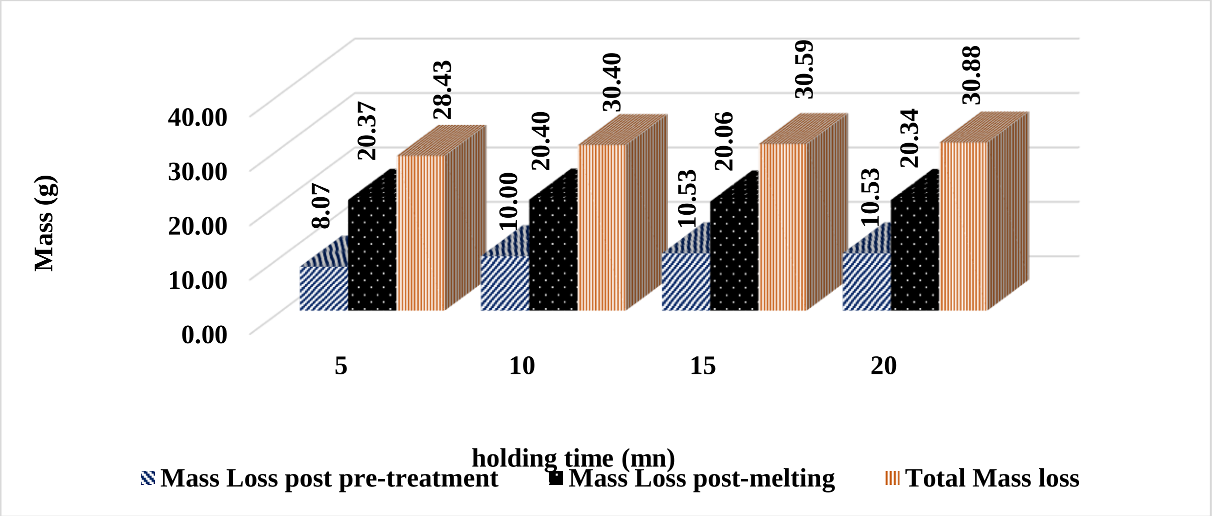


Figure 6. *Variation of mass loss with holding time*

3.3 Analysis of residues and slag

The results obtained from X-ray fluorescence (FX) analysis (Table 4) of the residues and slag of the treated samples clearly show that the longer the thermal refining time, the more the concentration of copper in the residues increases (Figure 7). Conversely, this concentration decreases in the slag. This relationship between the holding time of thermal refining and the concentration of copper in residues and slag reveals the crucial importance of this time in the recycling process.

It should be noted that recycled copper scrap often contains various winding wires of different dimensions. In some cases, these wires can constitute all of this raw material (Figure 8). Smaller diameter wires feature increased flexibility, making them particularly suitable for complex winding conFigureurations. These enameled wires are rated by their ability to withstand specific temperatures without degradation, with common classes including 403K, 428K, 453K and 493K. This classification allows them to be used in different environments with varying temperatures.

Therefore, the increased presence of copper in the residues is mainly attributed to the thermal degradation of small-sized winding wires. This degradation is visually justified by the clear presence of pieces of winding wires in the residues corresponding to holding times of 15 and 20 minutes (Figure 9). This observation highlights the importance of strategically managing holding time to improve the efficiency of the thermal refining process.

Table 4. *The X-ray fluorescence (XRF) analysis*

(a) Residues

	Holding Time(min)			
	5	10	15	20
Al2O3 (%)	1,31	1,38	1,38	1,39
CaO (%)	1,59	1,65	1,73	1,94
Fe2O3 (%)	0,75	1,30	1,81	1,85
K2O (%)	0,88	0,88	1,21	1,21
MgO (%)	1,05	0,83	0,16	0,9
MnO (%)	0,01	0,07	0,1	0,11
P2O5 (%)	1,23	1,88	3,16	3,21
S (%)	0,07	0,07	0,15	0,34
SiO2 (%)	1,33	1,33	1,52	1,51
TiO2 (%)	0,09	0,08	0,12	0,13
Cu (%)	58,78	59,11	65,27	81,44
Pb (%)	0,04	0,05	0,06	0,05
Zn (%)	0,07	0,13	0,19	0,25

(b) slag

	Holding Time(min)			
	5	10	15	20
Al2O3 (%)	3,17	1,97	1,38	1,23
CaO (%)	1,81	1,8	1,59	1,59
Fe2O3 (%)	1,81	1,73	0,95	0,95
K2O (%)	1,27	1,21	0,98	0,98
MgO (%)	1,05	0,71	0,44	0,43
MnO (%)	0,1	0,02	0,01	0,01
P2O5 (%)	1,88	1,72	1,16	1,14
S (%)	0,14	0,15	0,07	0,03
SiO2 (%)	10,19	8,87	6,08	6,05
TiO2 (%)	0,81	0,28	0,25	0,12
Cu (%)	37,18	35,12	35,08	35,13
Pb (%)	1,26	1,31	1,23	1,23
Zn (%)	1,09	1,13	1,18	1,1

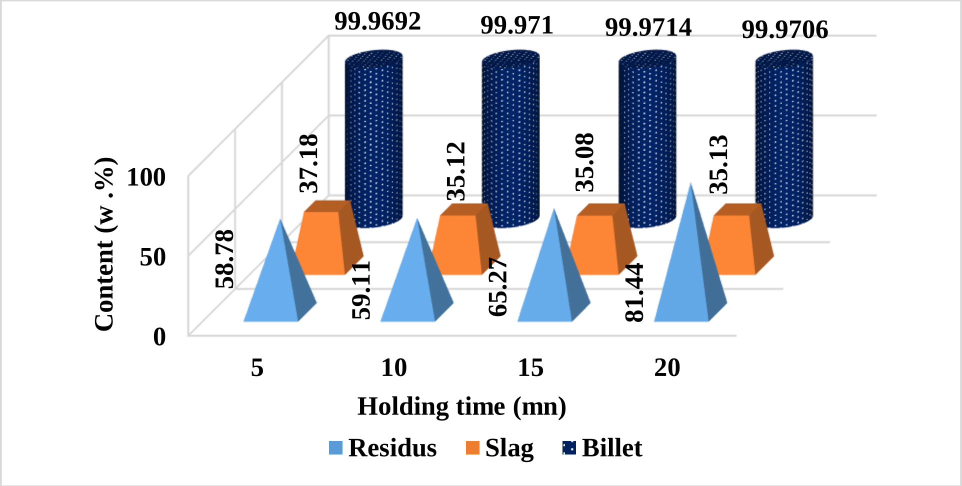


Figure 7. *Variation of Copper Content in Residues, Slags and billet with Holding Times*

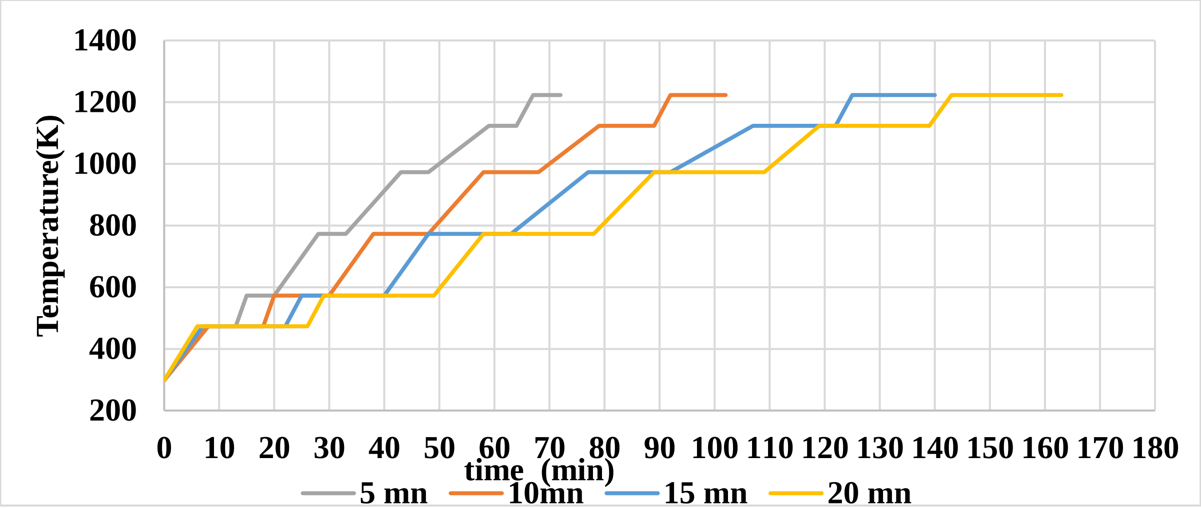
Figure 8. *Principal Constituents of copper scrap*

Figure 9. *Coil Wires in Residue of Holding Time 15 mn*

3.4 Thermal Refinement Time

For thermal refining with equal holding times, the 15 mn hold produces the highest copper content of 99.9714 .wt% (Figure 8). This represents an increase of 0.004% compared to the content obtained with the 10 mn hold , and 0.008% compared to the content obtained with the 20 mn hold. For this copper concentration, the time required for its thermal refining is estimated at 140 mn. This time compared to the times of those of 10 mn and 20 mn holding time, respectively generates an increase of 37.25% and a decrease of 14.12% (Figure 10 a).

As mentioned previously, thermal refining with a 15 min holding time caused significant copper loss due to wire degradation. To solve this problem, we tested four thermal refinings with different holding times (Figure 10 b,c,d et e). Compared to the 10 min refining, three of these thermal refinings with hold sequences of 5x2/10x2/15x2, 5x1/10x2/15x2/5x1 and 5x1/10x1/15x2/10x1/5x1 min gave almost similar results. These results are characterised by a slight increase in time from 2 to 3 min and a slight decrease in copper concentration of 0.0007%. However, the last one with a holding sequence of 5x2/10x2/15x1/5x1 proved to be particularly interesting. It delivered equivalent performances in terms of mass loss of residue and slag .Additionally, it features a significant reduction in refining time of 8 minutes and an increase in copper concentration reaching 99.9716% by weight with a 0.0006% increment.


Temperature(K)
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time (min)
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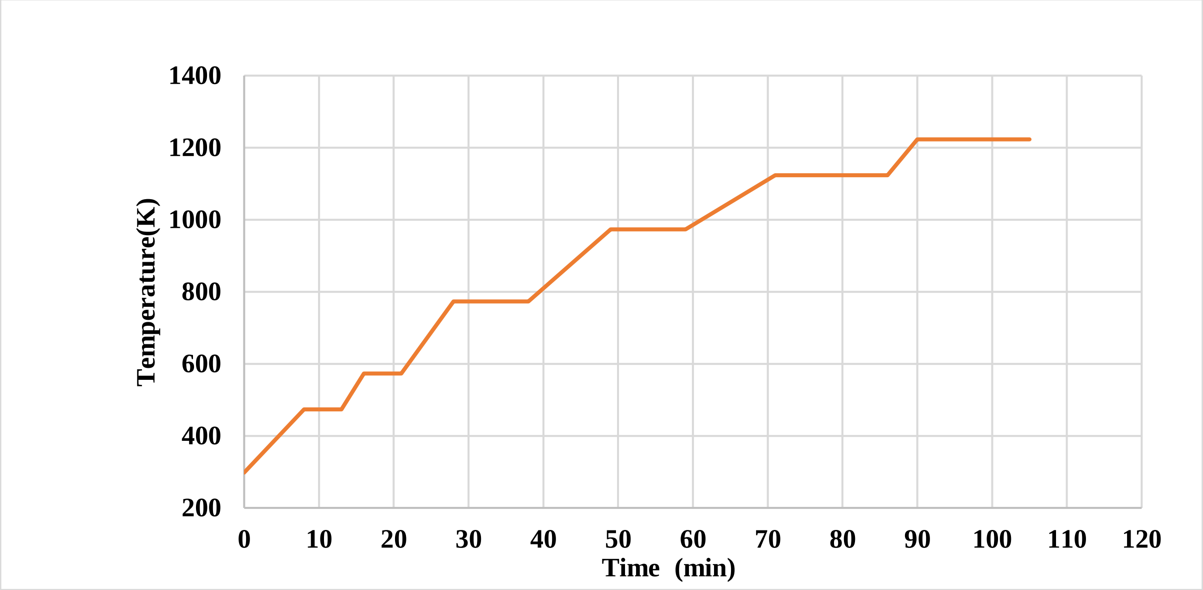
(a)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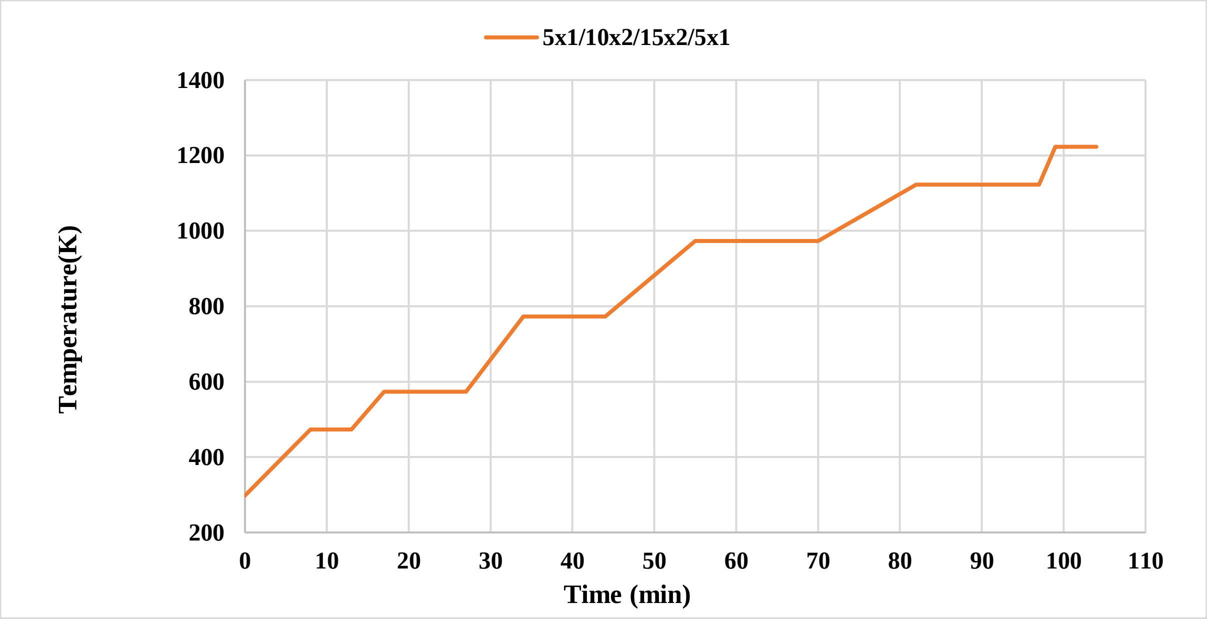
Time (min)

(b)


5x1/10x2/15x2/5x1
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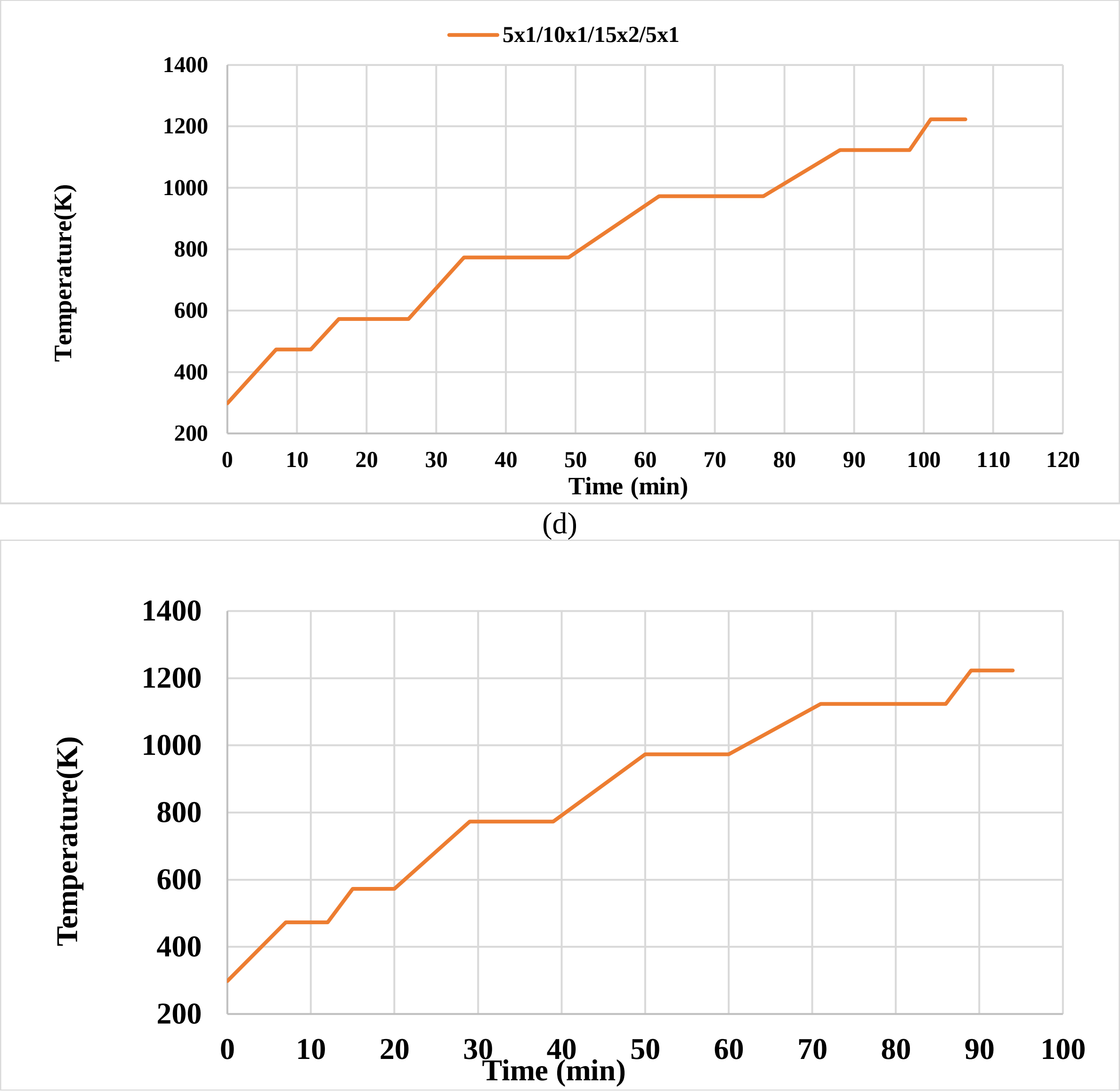
0 10 20 30 40 50 60 70 80 90 100 110
Time (min)

(c)



(d)

(e)

Figure 10. *Thermal refining type*
(a) Equivalent duration holding times (b): Sequence 5,5,10,10,15,15
(c) : Sequence 5,10,10,15,15,5 (d): Sequence 5,10,15,10,5 (e): Sequence 5, 5, 10, 10, 15, 5

The results of X-ray fluorescence analysis of residues from thermal refinements with varying holding times demonstrate clearly their effectiveness in minimizing the presence of copper in the treated sample residues (Table 5a). This reduction in copper content has significantly contributed to the results obtained by these refining processes in terms of purification (Figure 11).

Table 5: *X-ray Fluorescence (XRF) Analysis Results for Thermal Refinements with Varying Holding Times*

	Sequence			
	5x2/10x2/15x2	5x2/10x2/15x1/5x1	5x1/10x2/15x2/5x1	5x1/10x1/15x2/10x1/5x1
Al2O3 (%)	1,38	1,38	1,37	1,37
CaO (%)	1,74	2,04	1,75	1,76
Fe2O3 (%)	1,32	1,29	1,29	1,32
K2O (%)	1,16	1,09	1,11	1,16
MgO (%)	1,01	1,05	1,01	1,01
MnO (%)	0,07	0,07	0,07	0,07
P2O5 (%)	1,83	1,86	1,91	1,96

S (%)	0,09	0,07	0,09	0,09
SiO2 (%)	1,29	1,37	1,32	1,38
TiO2 (%)	0,09	0,09	0,09	0,08
Cu (%)	59,34	58,97	59,18	59,41
Pb (%)	0,05	0,05	0,06	0,06
Zn (%)	0,16	0,18	0,16	0,16

(a) Residus

	Sequence			
	5x2/10x2/15x2	5x2/10x2/15x1/5x1	5x1/10x2/15x2/5x1	5x1/10x1/15x2/10x1/5x1
Al2O3 (%)	1,91	2,44	2,18	1,91
CaO (%)	1,73	1,87	1,77	1,62
Fe2O3 (%)	1,62	1,82	1,73	1,62
K2O (%)	1,12	1,33	1,03	1,13
MgO (%)	0,66	1,01	0,73	0,64
MnO (%)	0,02	0,09	0,01	0,02
P2O5 (%)	1,34	1,83	1,2	1,67
S (%)	0,11	0,1	0,09	0,09
SiO2 (%)	7,17	8,51	8,48	8,48
TiO2 (%)	0,42	0,79	0,33	0,25
Cu (%)	36,02	35,03	36,01	35,14
Pb (%)	1,27	1,27	1,27	1,27
Zn (%)	1,12	1,19	1,13	1,13

(b) slag

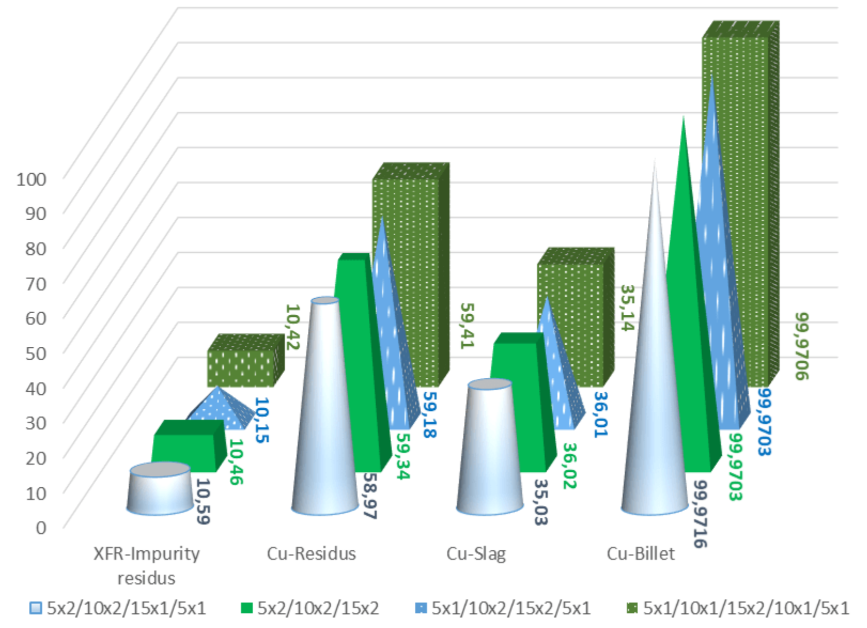


Figure 11. *Quantitative data on thermal refinements with variable holding times*

3.5 Thermal and Electric conductivity

The thermal conductivity tests were carried out on a small copper disc with a diameter of 25 mm and a thickness of 3.04 mm. This disc was machined from the copper billet resulting from the melting of the sample, which had undergone a thermal pre-treatment in the sequence 5x2/10x2/15x1/5x1. The results obtained using the H112A linear thermal conduction module showed a minimum thermal conductivity value of approximately 350 W/m-K [31].

Table 5. *Experimental values for billet cast from sample with pre-treatment in sequence 5x2/10x2/15x1/5x1*

	Values				
U(V)	42	55	68	70	91
I(Amps)	0,042	0,055	0,068	0,07	0,092
T1 (K)	25,6	28,2	31,4	32,5	39,9
T2 (K)	25,2	27,5	30,3	31,3	38
T3(K)	24,7	26,8	29,3	30,1	36,1
T6(K)	22,4	23	23,7	24,1	26
T7 (K)	22	22,3	22,8	23,1	24,4
T8(K)	21,7	21,7	21,9	22,1	22,9

Table 6. *Thermal conductivity for billet cast from sample with pre-treatment in sequence 5x2/10x2/15x1/5x1*

ΔX (m)	1,80E-02	1,02E+00	2,02E+00	3,02E+00	4,02E+00
A(m²)	4,906E-04	4,906E-04	4,906E-04	4,906E-04	4,906E-04
$\dot{Q}$ (W)	17,64	30,25	46,24	49	83,72
T_{hot} (K)	24,45	26,45	28,8	29,5	35,15
T_{cold} (K)	22,6	23,35	24,15	24,6	26,8
ΔT (K)	1,85	3,1	4,65	4,9	8,35
$\Delta T/\Delta X$ (K.m^{-1})	102,549889	171,84035	257,76053	271,61863	462,86031
K (W.m^{-1}.K^{-1})	350,601453	358,79844	365,63834	367,69427	368,66304

By applying the Wiedemann-Franz law ($K=L_0T/\rho$) at a temperature of 293 K [32-33], the electrical conductivity (ρ) of copper was estimated to be approximately 55.1 × 10^6 Ω^-1.m^- 1, an improvement over previous values reported in the study [26].

These results, which are consistent with those mentioned in the study[34], highlight the quality of the purification process tested. Pre-treatment with the 5x2/10x2/15x1/5x1 sequence considerably improved thermal and electrical conductivities. This improvement extends the possibilities for industrial use of the copper produced, particularly in the manufacture of certain types of electrical wire.

4. Conclusion

Comparing the copper content of the untreated sample billet (99.9230 .wt%) with that of the sample billet subjected to thermal refining with a holding time of 15 mn (99.9714 .wt%) , it is clear that thermal refining has significantly improved the purity of copper. The difference of 0.0484 wt% between the two concentrations highlights the effectiveness of the thermal refining process in removing impurities and increasing the copper concentration. This significant improvement demonstrates the potential of thermal refining to improve the quality of molten copper and highlights its importance in the purification process by exploiting the volatility of some impurity elements and the oxidation of others to slag under ambient conditions.

However, it is crucial to note that significant mass losses occur during thermal refining of 15 min holding time due to degradation of small wires. This loss increases with temperature for thermal refinements with equivalent holding times. To alleviate this problem, implementing thermal refinement with varied holding times presents itself as a viable solution. This is exemplified by thermal refining with a hold sequence 5x2/10x2/15x1/5x1 min, which not only increased the copper content to 99.9716 wt%, but also improved the electrical conductivity and thermal by reducing the total pre-treatment time to 94 minutes. This approach provides a more effective solution for optimising both the purity of the copper and the operational efficiency of the recycling process, while producing a product with a wide range of industrial applications.

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