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Trusted and Transparent Blockchain-enabled E-waste Optimization to Recover Precious Metals with Microwave Heating

Rajendra Prasad Mahapatra[1], Satya Sai Srikant[1]*, Sachi Pandey[1], Vikas Chouhan [2] and Raghupatruni Bhima Rao[3]
[1]SRM Institute of Science and Technology, Delhi – NCR Campus, Modinagar, India 201 204
[2]Indian Institute of Technology, Roorkee, India 247 667
[3]CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, India 751 013

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Blockchain technology facilitates trust and transparency in the decision-making process and enables the transaction's verifiability by reading immutable distributed ledgers. It has been innovatively applied this technology in the E-waste optimization for the recovery of precious metals using microwave heat treatment. This present paper presents the maximum recovery of precious and base metals from E-waste with a numerical technique called surface response methodology, and was compared with the actual experimental results. The main goal of this paper is to recover the precious metals like copper and gold with its adjacent metals from unwanted and discarded printed circuit boards, integrated circuits, and standards connectors, with the input variables of microwave power, maximum temperature, and aqua leaching ratio. The obtained empirical information of recovered metals was recorded in immutable distributed ledgers so that every member of the blockchain network can be read and verified through the stored records. These records were also utilized to minimize the error and maximize the precious metal outcomes. The result with blockchain network also shows that identical resemblance between the experimental and statistical predicted data obtained with surface methodology. Further, Smart Contracts has been created and deployed to store and retrieve empirical records in the Hyperledger Fabric Blockchain Platform and then measured the performance using Hyperledger Caliper Benchmark.

Keyword: Aqua leaching, Smart contract, Surface response methodology, Temperature

Introduction

In the present generation, the production and its consumption of electric-electronic components substantially increased. Within the last decade, the life span of any consumable electronics, electric equipment (e.g., computers, mobile phones, printed circuit boards) decreased from 5 years to 2 years or even less time. The life period of such materials become waste, which is to be disposed of and hence called as electronic and electrical waste.[1,2] Recycling of E-waste is important because it contains precious metals content in it and avoids environmental pollution due to the continuous disposal of waste such as landfilling, dumping, etc. Therefore, E-waste optimizations have been the subject of active research from the last decades, intending to maximize the recycling and reusability of waste materials. To maximize the trust and transparency of metal recovery, we create and deploy Smart Contracts (SC) in the blockchain platform that records every experimental detail in a block in the form of a transaction after approval from the network endorsement peers.[3,4]

The endorsement peers verify and validate the legitimacy of the transaction as per the pre-defined endorsement policies. Block is added to the chain of shared distributed ledger.[4] This chain is identical throughout the network and tamper-proof. The blockchain controlled in a distributed manner by multiple entities, and it does not have a single point of failure.[5] The Smart Contracts (SC) is a protocol used to create transactions, and that is automatically executed when the event is triggered.

The recovery of precious metals from e-waste through hydrometallurgical processes along with heating is much economical than any other methods but this method is environmentally polluted, hence not recommended as an eco-friendly method.[6] As per available literature so far, the recovery of precious metals from e-waste can be performed either by muffle furnace heating followed by leaching or plasma-treated coupled with leaching. However, very few researchers have attempted to use the microwave

*Authors for Correspondence
E-mail: satya.srikant@gmail.com ; satyas@srmist.edu.in

technique on E-waste, followed by a leaching process with various techniques.

The results seem to be very positive, useful, and clean, as the microwave technique recovers the precious metals obtained in much lesser time and also lesser temperature with selective heating as compared to conventional and plasma methods. Due to the above reasons, the authors started investigating the effect of judicious combinations of microwave heat treatment and leaching processes on the recovery of precious metals from e-waste,[7,8] with wonderful results of having around 93% of copper and around 14% of gold with 50–100 Kg of e-waste samples. Furthermore, these experiments were investigated several times repeatedly by increasing the e-waste samples, and similar results were obtained at each investigation. It was necessary to optimize the process variables to recover maximum precious metals from E-waste with an environmentally friendly method. In lieu of this, design optimization was investigated from the strategic mathematical methodology. This mathematical methodology is known as surface response methodology. It helps in predicting the maximum recovery of precious metals with the given variables and hence optimizing the process parameters. This methodology has already been applied in the optimizing of process parameters in many applications like grinding experiments of coal samples[9], producing the graphite concentrates[10], upgradation of sillimanite[11], and optimization model for the separation of titanium from beach sand minerals.[12,13]

In this present investigation, anunique attempt has been made with optimization with surface response methodology, which consists of Design expert 6.0.6 (ANOVA) simulator and MATLAB 8.1alongwith Blockchain concept. The statistical results obtained with response methodology were compared with the experimental results obtained with the judicious combinations of microwave technique and aqua-acid leaching from powdered E-waste.[7,8] This investigated empirical information is then reflected in the blockchain via Smart Contacts (SC). The SCs are implemented in Go language and deployed in Hyperledger Fabric (HF) platform, and performance is measured using the Hyperledger Caliper benchmark tool.

Materials and Methods

Raw Material and Microwave Heating

The electronic materials like electrolyte capacitors, batteries, small transformers, and plastics from E-Waste samples of printed circuit boards (obtained from the scrapped old computer and mobiles) were removed first. Some metal wires and epoxy base plates were removed with sharp tools like cutter and nose pliers. The PCB samples with integrated chips (ICs) and pogo pins after dismantling / removal by cutter, were also the raw materials for our investigations. These samples were first shredded into fragments of approximately 10 mm × 10 mm and finally grinded into finer particles of an approximate size of 250–300 µm (i.e., 50–60 mesh) in the laboratory. These grinded E-waste materials of around 50 Kg were kept in a quartz container inside the microwave furnace for heating. The heated sample with the power of 2 KW produces a temperature of around 1100ºC. The heating process got further enhanced either directly or indirectly with the help of silicon carbide (SiC) susceptor and spreading the silicon carbide (SiC) powder, as these carbon materials behave as a coupling agent.[14–17] The placement of crushed E-waste sample in a microwave furnace was very important for volumetric heating, and hence authors have decided the placement of the sample as shown in Fig. 1 based on their experiences.

Aqua-Leaching Process

The heated sample was carried out for the acid leaching process with concentrated hydrochloric acid (HCl). After the leaching process, all sample material along with dismantled metallic elements /pins, integrated chip, transistors, diodes, RAM, undergoes the aqua regia process. The Aqua regia process is performed with the mixtures of hydrochloric acid (HCl) and nitric acid (HNO_3) in the ratio of 3:1 for 2 hours.[7,8]

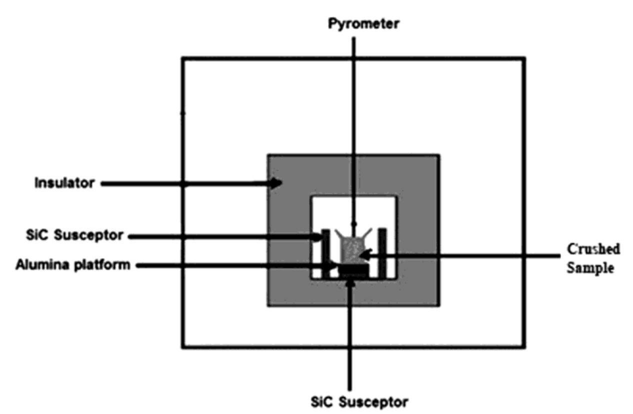

Fig. 1 — Placement of crushed e-waste sample inside the microwave furnace for microwave treatment

Surface Response Methodology

It is desired to study the analysis and optimization for the output responses, i.e., the recovery of copper and gold from the “aqua-leached microwave-heated e-waste grinded sample”. The variable such as microwave temperature and aqua leaching (ratio) were varied accordingly, keeping other parameters microwave power, and leaching as constant. These variables were decided as per the experimental design for microwave heating of E-waste followed by the leaching process. The variable for aqua leaching at different levels is shown in Table 1.

In the present study, the Box-Behnken factorial design was chosen to find out the relation between the response functions (Cu % recovery and Gold % recovery) and three variables (microwave power, maximum temperature, and the ratio of aqua leaching). Let the three variables be defined as:

A = Microwave Power (W)

B = Temperature (°C)

C = Aqua Leaching (Ratio)

The results are evaluated by using the Box–Behnken factorial design. To optimize the quantity and amount of copper and gold from "aqua-leached of microwave heated e-waste crushed sample", the complex mathematical equations are formulated with the help Box–Behnken based Design Expert 6.0.6-ANOVA software and MATLAB.

The responses of all designed experiments were analyzed by using Design Expert 6.0.6- ANOVA software in which the design class and type of experiment were chosen in the first step. In this investigation, the Box-Behnken factorial design was selected, followed by its surface response. Then the designs were made for various factors like maximum power, temperature, and aqua leaching, with units and their values from minimum to maximum values and also their responses for various precious metals recovery. Minimum experiments of 13 sets with variables at different conditions and their responses were recorded and analyzed in the form of 3-dimensional response surface graphs that illustrate the effects on the outputs of changing the input variables' values. If all the variables were assumed to be measurable, then its response surface can be expressed as:

$$V_o = f\,(u_1, u_2, u_3 \ldots u_K) \qquad \ldots (1)$$

Here V_o is the output and u_K the variables of action called factors. For the Box-Behnken’s three-level factorial experimental designs, a total of thirteen experimental runs were performed. The objective was to optimize the response (with an assumption that the independent variables are continuous and controllable through the experimentation. It was also required to find a suitable approximation for the true functional relationship between independent variables and the response surface. A complete description of the process behavior of a quadratic model:

$$V = b_0 + b_1u_1 + b_2u_2 + b_3u_3 + b_{11}u_1^2 + b_{22}u_2^2 + b_{33}u_3^2 + b_{12}u_1u_2 + b_{23}u_2u_3 + b_{13}u_1u_3 \qquad \ldots (2)$$

Generally, Equation 2 can be written in matrix form

$$V = bU + \varepsilon \qquad \ldots (3)$$

where V, defined to be a matrix of measured values, U to be a matrix of independent variables. The matrices b and ε consists of coefficients and errors, respectively. The solution of Equation 3 can be obtained from the matrix approach

$$b = (U'U)^{-1}U'V \qquad \ldots (4)$$

Here U ' shows the transpose of the matrix U and $(U'U)^{-1}$ is the inverse of the matrix U ' U.

Table 1 — Variables of Aqua Leaching at different levels of ratio

Variables	Levels with ratio 3:1		
	Low (-1)	Centre (0)	High (+1)
Aqua Leaching Ratio	3.00:1	3.75:1.25	4.50:1.50
Microwave Power (Watt)	2900	3000	3100
Maximum Temperature (ºC)	1400	1450	1500

Smart Contracts

Several Smart Contracts (SC) were created and deployed in the blockchain platform. It has been focused especially on the two specific contracts. Firstly, it provides the record creation and secondly it observes continuously, based on the various experimental inputs’ parameters. Initially, three structures have been defined as shown in Table 2 to collect the required information. The first 'Input' structure contains the required input information, e.g., maximum temperature, aqua leaching ratio, methodology, and type of E-waste material. The

Table 2 — List of Structures

For Initialize Input Values

Input {Max Temp string, Aqua Leach Ratio string, Methodology string, Ewaste Material Type string}

For Setting Metal Accuracy

Metal Accuracy {Cu string, Gold string}

For Creating Record

Record {RID string, Input Values Input, Achieved Metal Accuracy} second 'Metal Accuracy' structure is defined for storing the accuracy of both Cu and Gold. The third structure creates 'Record' with key RID and contains both 'Input' and 'Metal Accuracy' structure. The final records were stored in a Blockchain corresponding to the key RID.

The record was created and written on the decentralized platform using Smart Contract, as described in Algorithm 1. It takes stub and a list of arguments as an input that returns a corresponding response. This contract was first initialized for all the variables of structures took from the input arguments. It checks in the next step, whether the record exists otherwise it returns an error. Later, it marshals the record and stores them into the block corresponding to the record ID by executing the function PutState. Finally, it returns a successful response containing a message “record has been stored”.

Algorithm 1

```
Input   : stub ChaincodeStubInterface, args [ ]string
Output : Response
begin
    /* Initializing all the structures                    */
    var input Input
    input.MaxTemp :⟵ InitStructValue(args[1])
    input.AquaLeachRatio :⟵ InitStructValue(args[2])
    input.Methodology :⟵ InitStructValue(args[3])
    input.EwasteMaterialType :⟵ InitStructValue(args[4])
    var metalaccuracy MetalAccuracy
    metalaccuracy.Cu :⟵ InitStructValue(args[5])
    metalaccuracy.Gold :⟵ InitStructValue(args[6])
    var record Record
    record.ID :⟵ InitStructValue(args[0])
    record.InputValues :⟵ InitStructValue(input)
    record.AchievedAccuracy :⟵ InitStructValue(metalaccuracy)
    if IsRecordExist(stub, record.ID) then
        return Error("record " + record.ID + "is already exists.")
    end
    /* Putting state in block                             */
    PutState(record.ID, Marshal(record))
    /* record has been entered                            */
    return Success("record has been stored")
end
```

Algorithm 2 represents the smart contracts (SC) described about the procedure of retrieving a stored record from the decentralized platform. This contract takes stub and arguments as an input and returns the retrieved record from blockchain corresponding to the input record identity RID as an argument. First, it invokes the GetState method of the blockchain's world state database to retrieve stored information as a Bytes form (recordAsBytes) if information exists; otherwise, it failed to get state and written an error response. Then the record variable is created to store the unmarshal information of recordAsBytes. Finally, it returns a success response, including retrieved information.

Algorithm 2

```
Input   : stub ChaincodeStubInterface, args [ ]string
Output : Response
begin
    rid :⟵ args[0]
    recordAsBytes, err :⟵ stub.GetState(rid)
    if err != nil then
        jsonResp ⟵ "{\"Error\" : \"Failed to get state for" + rid + "\"}"
        return Error(jsonResp)
    end
    var record Record
    Unmarshal(recordAsBytes, &record)
    return Success(recordAsBytes)
end
```

Results and Discussion

As shown in Table 2, the mathematical model for copper and gold recovery from microwave heated E-waste crushed sample was designed on the basis of experiments investigated with maximum temperature and aqua leaching as input variables. The microwave power and leaching were kept constant through entire investigations. The aqua leaching is basically obtained by mixing hydrochloric acid (HCl) and nitric acid (HNO_3) in the ratio of 3:1 for 2 hours. It has been observed that recovery of gold was not proper with the aqua leaching process in the ratio of 3:1, so it was experimented to see the possible effect by increasing the ratio of hydrochloric acid (HCl) and nitric acid (HNO_3) in the ratio as 3.25:1; 3.25:1.25; 3.50:1.25 and 3.75:1.25 as shown in Table 3.

Predicted Recovery of Copper and Gold

The mathematical equation obtained from ANOVA for Cu% recovery (with respect to Fig. 2) and Gold% recovery (with respect to Fig 3) from the E-waste sample are shown in Eqs 5 & 6, respectively, along with their corresponding R square values. In Table 4

Table 3 — Recovery of precious metals from E-waste with Microwave power maintained at 3000 Watt, Max Temp (ºC) and Aqua Leaching (Ratio) as main variables

Exp. No	Input		Recovery (Responses)	
	Max Temp (°C)	Aqua Leach (Ratio)	Cu (%)	Gold (%)
1	1450	3:1	75	1.3
2	1450	3.25:1	89	2.4
3	1440	3.25:1.25	87	1.25
4	1430	3.50:1.25	84	3.2
5	1440	3.75:1.25	93	14.31
6	1435	3.75:1.50	91	1.56
7	1450	4.50:1.50	87.19	6.2

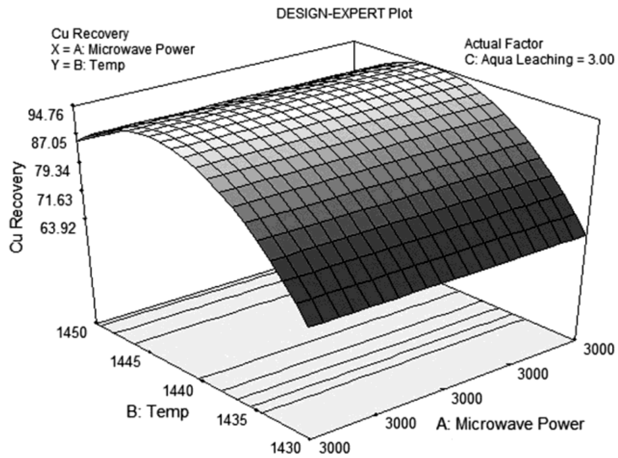

Fig. 2 — Recovery of Copper % from aqua-leached of microwave heated e-waste ground sample

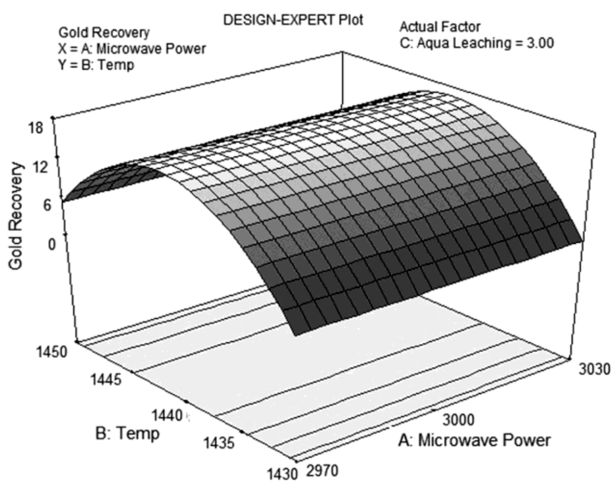

Fig. 3 — Recovery of Gold % from aqua-leached of microwave heated e-waste ground sample

is shown the predicted recovery of copper and gold with variation of temperature from the micro wave heated E-waste sample (for given maximum aqua leach ratio i.e 3.75:1.25 at maximum power 3000W). There were some other factors like R square (R^2), adjoint R square (adj.R^2) and standard deviation values for predicted recovery of copper and gold, as shown in Table 5.

Cu % recovery = 94.99 + 33.44 * B −10.45C

− 260.34 B^2 −19.38 C^2+ 124.88 BC … (5)

R^2 value for copper recovery = 93.27 %

Gold %recovery = 17.31 + 43.36 * B − 12.56C

− 326.87 B^2 − 34.83 C^2 + 163.05 BC … (6)

R^2 value for gold recovery = 87.39 %

Table 4 — Recovery responses from E-waste obtained with ANOVA 3-D curve with optimized Aqua Leach ratio 3.75:1.25=3 at 3000 W power

Sr. No.	Input	Recovery (Responses)	
	Max Temp (°C)	Cu (%)	Gold (%)
1	1430	64.21	0.0
2	1435	70.58	3.6
3	1438	92.89	13.82
4	1445	92.47	11.98
5	1450	85.87	5.24

Table 5 — ANOVA responses for copper and gold recovery

Recovery	Responses (ANOVA with Box Behnken Design)				
	Predicted Yield (%)	R^2 (%)	Adj R^2 (%)	Std Dev	Std Err (%)
Copper	92.89	93.27	92.32	3.36	0.01
Gold	13.82	87.39	86.87	2.39	3.45

Table 6 — Experimental and predicted value of copper and gold

Analysis	Input		Recovery	
	Temp (°C)	Aqua Leach (%)	Cu (%)	Gold (%)
Actual	1440	3.75:1.25 = 3	93	14.31
Predicted	1438	2.998	92.89	13.82

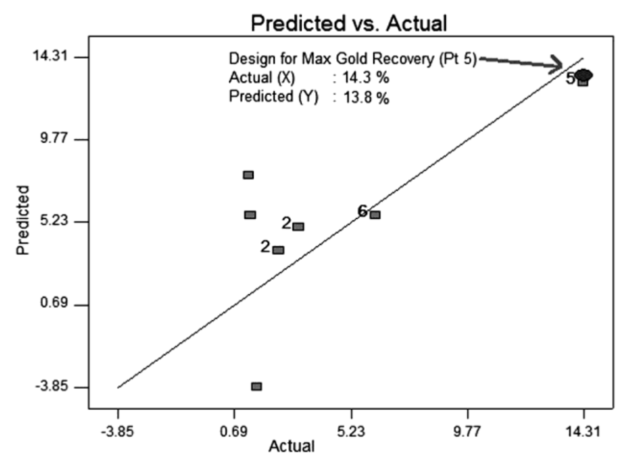

Fig. 4 — Predicted and experimental gold % recovery from present investigations

However, the entire investigations were carefully studied for optimized and predicted gold % recovery shown in Fig 4 and Table 6.

It is observed from Table 6 that the experimental and the statistical predicted data for optimized recovery of copper and gold are matching, and it contains very minimal error (i.e., 3.45%) for gold recovery and 0.01% error for copper recovery.

The block chain implementation work was verified through the Caliper benchmark testing to meet the expected performance of deployed Smart Contracts (SC). The performances of both the SCs are measured on the basis of success and failure rate, transaction

```
2020.09.14-23:42:33.963 info  [caliper] [round-orchestrator]    Finished round 2 (Get Record.) in 1.997 seconds
2020.09.14-23:42:33.964 info  [caliper] [report-builder]        ### All test results ###
2020.09.14-23:42:33.969 info  [caliper] [report-builder]
+----------------+------+------+-----------------+-----------------+-----------------+-----------------+-----------------+
| Name           | Succ | Fail | Send Rate (TPS) | Max Latency (s) | Min Latency (s) | Avg Latency (s) | Throughput (TPS) |
|----------------|------|------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Create Record. | 100  | 0    | 51.2            | 6.27            | 1.92            | 4.26            | 14.9            |
|----------------|------|------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Get Record.    | 100  | 0    | 51.0            | 0.09            | 0.01            | 0.03            | 50.3            |
+----------------+------+------+-----------------+-----------------+-----------------+-----------------+-----------------+

2020.09.14-23:42:34.004 info  [caliper] [caliper-flow]  Generated report with path /hyperledger/caliper/workspace/report.html
2020.09.14-23:42:34.106 info  [caliper] [message-handler]       Handling "exit" message
2020.09.14-23:42:34.108 info  [caliper] [round-orchestrator]    Benchmark finished in 30.385 seconds. Total rounds: 2. Successful rounds: 2. Failed rounds: 0.
2020.09.14-23:42:34.108 info  [caliper] [caliper-engine]        Network configuration attribute "caliper.command.end" is not present, skipping end command
2020.09.14-23:42:34.109 info  [caliper] [cli-launch-master]     Benchmark successfully finished
vikas@TheDevice:~/3Caliper/BasicNetwork_1.4.4$
```

Fig. 5 — Summary of Smart Contracts Performance Metrics

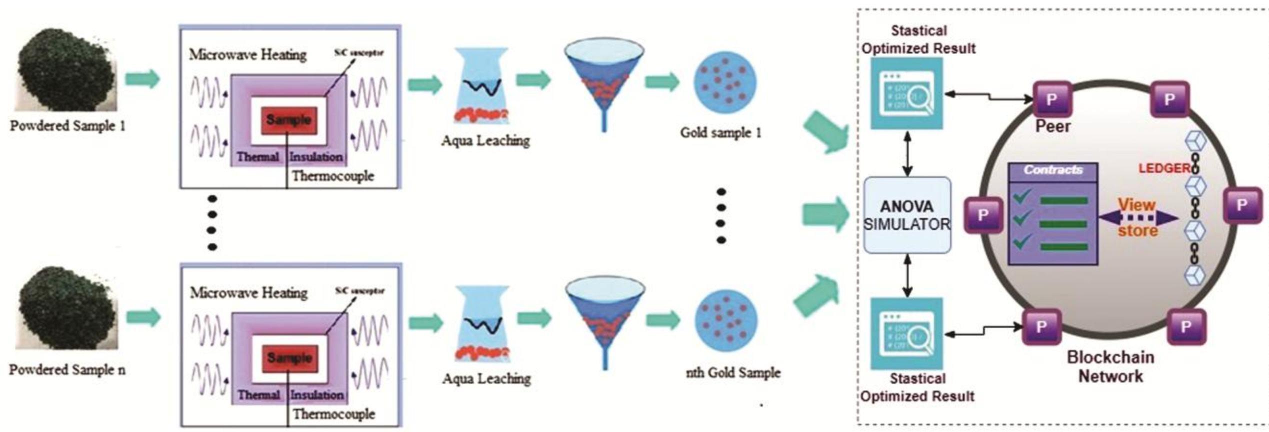

Fig. 6 — Graphical representation of entire investigations

latencies, and the achieved throughput in transactions per second (tps). Two modules are tested on the block chain platform. Firstly, the record was written into block chain and secondly, the retrieving stored was recorded.

The screenshot of the Caliper benchmark performance metric as shown in Fig 5 showed that both the contracts ran and worked successfully without any failure or timeouts. The type of rate control was pre-defined to the fixed-rate with 50 tps in the present investigations. Therefore, the sending rate was around 50 for both the contacts. Further, the contact's latencies were recorded in seconds; however, the record creation contact took a little more second because it involved an additional operation such as information collection and marshalling. The throughput was depended upon the latency; therefore, the throughput of the first test was 14.9 tps, and the second test was 50.3 tps. Based on results, it was concluded that the retrieving modules are faster than the writing modules. The process of the entire investigations can be shown in Fig 6 graphically.

Conclusions

The present investigations were targeted for the construction of trusted and transparent E-waste optimization in a verifiable manner using Blockchain technology. The experimental records were designed in the blockchain to avoid asymmetry in the achieved experimental results, and these records can be utilized to minimize efforts in deciding experimental inputs and maximize the metal outcomes. The following conclusions were drawn from the experimental and optimization predicted studies carried out for the optimized recovery of copper and gold from aqua-leached of microwave heated e-waste crushed sample.

- ❖ It has been observed that recovery of optimized gold was obtained with the aqua leaching process in the ratio of 3.75 : 1.25 instead of the exact 3:1 ratio.
- ❖ The three factorial ‘Box Behnken Design’ with response surface methodology employed successfully for optimized recovery of copper and gold. Different equations are developed with varying two parameters, i.e., temperature and percentage of aqua leaching, keeping microwave power constant.
- ❖ The output response obtained with response surface methodology showing 92.6% of copper and 13.82% of gold. In order to accomplish a better understanding of copper and gold recovery,

the predicted model values were represented in 3-Dimensional response surface graphs.

- It is clear from the study that the predicted responses for recovery of copper and gold from E-waste could be optimized using surface response optimization techniques, which closely resembles the experimental results.

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