# Effects of Flocculant, Surfactant, Coagulant, and Filter Aid on Efficiency of Filtration Processing of Copper Concentrate: Mechanism and Optimization

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| Keywords | Abstract |
|---|---|
| *Vacuum filter*<br><br>*Additives*<br><br>*Water recovery*<br><br>*Cake formation rate*<br><br>*Moisture content* | In the recent decades, water scarcity has become a major challenge for many reasons, especially the inadequate use of water resources. The mineral processing plant is among the most important water-consuming industries. Filtration, as one of the important processes in water recovery, is a process in which the solid-suspended particles are removed from the liquid. In the present work, the effect of the additives affecting the filtration process upon the responses including the resistance to filter cloth (R), specific cake resistance ($\alpha$), moisture content, water recovery rate, and cake formation rate by the vacuum top-feed method is investigated. The experiments are performed by two methodologies: one-factor-at-a-time and statistical analysis. The additives are the flocculant, coagulant, surfactant, and filter aid. According to the one-factor-at-a-time methodology, the optimal type and dosage of the variables are as follow: flocculant A25 with a concentration of 15 g/L, perlite as the filter aid with an amount of 2.5%, surfactant cop 20-101 with a concentration of 3 cc/L, and the coagulant $CaCl_2.2H_2O$ with a concentration of 2.5 g/L. The usage of the flocculant, surfactant, and filter aid at the same time is also statistically analyzed with the aim of maximizing the cake formation rate and minimizing the moisture content of the filter cake. Under the optimal conditions and taking into account 11.68 g/t of the flocculant A25, 3.8% of perlite as the filter aid, and2.92 cc/L of the surfactant cop 20-101, the cake formation rate and the moisture content were obtained to be 0.297 mm/s and 12.7 %, respectively. |

## 1. Introduction

### 1.1. Filtration theory and fundamental aspects

Water usage in the mining industry is common, and without the presence of this critical substance, it will be impossible to perform the mining activities. Water is one of the most important resources in the world since life cannot exist and industry cannot operate without it. Domestic and industrial usages of water generally produce deterioration in its quality and, in most instances, wastewater must be collected and given effective treatment before being released into the environment. In many cases, treated wastewater may be available for re-use within certain systems in industrial operations [1]. Water is widely used in mineral processing plants and hydrometallurgical processes. Due to the global increase in metal production, the demand for water including freshwater is expected to increase steadily [2]. Water shortage in arid and semi-arid regions is one of the most important issues for these areas because water is essential for the development of all economic activities, environmental care, and quality of social life. The mining industry is critical to the logical and efficient usage of water in its activities. For example, water scarcity in Chile's mineral processing plants has led to the usage of seawater for processing operations [2, 3]. The main technique involved in dewatering processes is

sedimentation, which may include gravity, centrifugal force or any other force [4]. Recycling of industrial wastewaters as well as mineral processing effluents is very crucial, in particular, to meet the requirement of potable, industrial, and agricultural water [5]. Two types of common solid-liquid separation processes in mineral processing include sedimentation and filtration [6]. Solid-liquid separation by mechanical means such as centrifugation, vacuum filtration, and pressure filtration is a common practice for sludge treatment. The volume of the sludge that contains as high as 99% of water can be considerably reduced by the mechanical dewatering process. The resulting solid product can then be transported, treated or disposed more easily [7]. The higher proportion of the water is first removed by the precipitation method in thickener to produce a high-density pulp with a solid content of about 45-65%. In some processing plants, up to 70% of water can be recovered at this stage [8, 9]. Filtration systems have been used in many industrial sectors such as chemical, nuclear, food, and mineral processing plants [10]. The filter consists of a porous layer whose pores are suitable for liquid passage but prevent the passage of solid particles [11, 12]. Filtration, a typical energy-saving non-thermal dehydration (liquid) unit operation that uses filtration media to separate solids and liquids, has attracted a wide attention in different industries. In the 20th century, with the rapid development of the three major chemical industries (coal, oil, and natural gas), many intermediate products are required to be filtered, dehydrated, and separated during production, thereby rendering the filtration technology an unprecedented development, and is now widely used in food, environment, mineral processing, and chemical industries [13].

Filtration has been categorized according to various aspects of filtration processes. There are, however, a number of different classifications depending on the authors. Ullmann's Encyclopedia of Industrial Chemistry offers a classification system based on four parameters. A short section from Ullmann is presented here [14].

Filtration processes can be classified in accordance with different criteria [14]:

      1. Location of particle retention
      2. Generation of the pressure difference
      3. Operation mode
      4. Application

      Vacuum filters are one of the most important and most used filters in the industries. A vacuum filter is a unit used for dewatering various minerals

such as calcium carbonate, phosphates, and sulfates. Continuous vacuum filters are usually used when solids are not fine. They have a high sedimentation rate and produce a permeable cake that can be carried out with a moderate pressure difference in the dewatering process [15, 16]. The sludge dewatering performance is evaluated by the filtration rate and cake solid content. The filtration theory has been developed for the evaluation of filterability of sludge over the years based on the pioneering work by Ruth [17]. This theory assumes 1D Darcian flow, no mass transfer between liquids and solids, insignificant gravitational forces, and a negligible solid velocity compared to the liquid velocity. The equation can be expressed as:

$$q = \frac{1}{\alpha\mu}\frac{dp_l}{d\omega} \tag{1}$$

where $q$ is the liquid flux (m/s) relative to the solids; $\mu$ is the liquid viscosity (Pa s); $\alpha$ is the local specific resistance (m/kg); $pl$ is the liquid pressure (Pa), and $\omega$ is the position in the cake in the form of the amount of solids deposited per unit cross-section area (kg/m$^2$).

The final equation used in the conventional filtration process is the following one:

$$\frac{dt}{dv} = \frac{\mu\omega\alpha_{av}}{PA^2}V + \frac{\mu R_m}{PA} \tag{2}$$

At the filtration stage, the average specific filtration resistance is constant; a plot of the inverse flux ($dt/dV$) against the filtrate volume ($V$) should be linear. Once the cake formation is complete and the compression stage begins, $dt/dV$ increases markedly with increasing filtrate volume. The plot is no longer linear with the slope sharply increasing, as shown in Figure 1. This change can be used to differentiate between filtration and compression [17].

According to the data recorded during the filtration tests, in addition to the specific cake resistance and the resistance to filter cloth, the other parameters involved are also calculated. One of these parameters is the average cake formation rate ($L_{gr}$), which is equal to the ratio of the cake thickness to the filtration time. This parameter is also used to select the type of filtration device.

$$L_{gr} = \frac{L_e}{t_e} \ or \ \frac{L_e}{t_{tr}} \tag{3}$$

In cases where the thickness of the cake is not measurable during the test, the thickness of the cake is calculated using one of the two equations below:

$$L_e \approx \frac{V_e \ \rho_L}{A \ \rho_s \left[\frac{1-S}{S} - (1-C_{av})\left(\frac{\rho_s(1-S)}{\rho_L \ S}+1\right)\right]} \approx \frac{S \ V_e \ \rho_L \left[\frac{\rho_s}{\rho_L}(m_{av}-1)+1\right]}{A \ \rho_s(1-m_{av} \ S)} \tag{4}$$
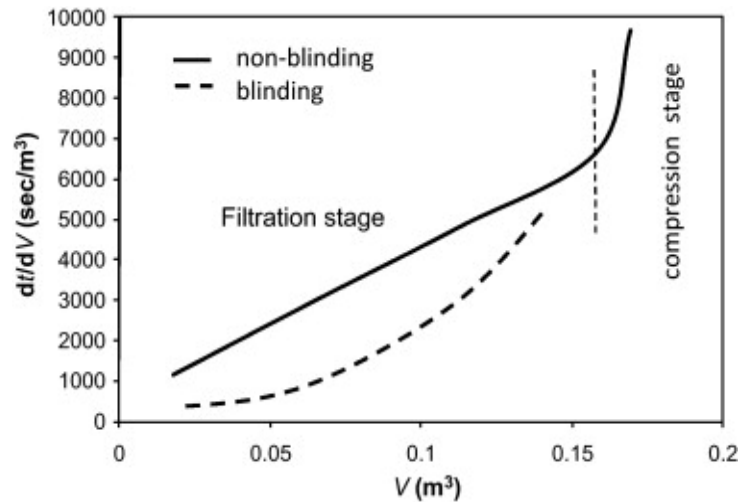
**Figure 1. Graph of t/v in terms of v [17].**

## 1.2. Influential variables

In general, the variables affecting the filtration process can be classified into three groups: the technical specifications of the filtration device used (such as the maximum difference between the pressures produced on both sides of the filtration media, filtration time, type of filtration media, device volume, and filtration area), the characteristics of the materials to be filtered (such as the pulp present, pulp temperature, pulp viscosity and specific gravity, pulp pH, particle size, and particle shape), and the type of material used to improve the performance of the filtration process that is called the filtration chemistry (such as the flocculant, coagulant, and filter aid) [18-20]. Patra *et al*. (2016) have studied and improved dewatering of the iron ore fines by the usage of surfactants. In their primary studies, they found that the main problem with the dewatering process was adherence of the ultra-fine particles to the iron particles. Surfactants improved dewatering by increasing the hydrophobicity. The surfactant cetyl trimethyl ammonium bromide (CTAB) was used to neutralize the particle charge. By adding CTAB, the moisture dropped from 12-13% to 9-10% [21]. Castro and Laskowski (2015) have studied the effect of flocculants on flotation of copper-molybdenum ore. The results obtained showed that the flocculant polyacrylamide (PAM) had a negative effect on the recovery. It was also found that the flocculent polyethylene oxide (PEO) was an effective one for molybdenum in a wide range of pH values but in any case affected the molybdenum flotation efficiency [22]. Fan *et al*. (2015) have studied the effect of particle properties

on the filtration of fine-grained coal pulp and the structure of cake formed. Their studies postulated that the addition of kerosene to the pulp led to an increase in the cake hydrophobicity and a decrease in the moisture content of cake to 4.41%. Addition of the flocculent to the pulp, due to bridging between the particles or neutralizing the particle surface charge, could form large clots so that the filter cake resistance decreased and the permeability increased [23]. Garmsiri and Shirazi (2014) have examined the effect of grain size on the preparation of flocculants. In this work, the effect of the size of solid flocculant particles, grain size reduction, and their solubility with high molecular weight anionic flocculant were studied. The results obtained showed that with smaller particles, less time was required to achieve a high settlement rate. The dissolution heterogeneity decreased due to the presence of gels by filtration. By increasing the grain size, the preparation time of the flocculant increased, and, on the other hand, reducing the amount of coarse grains led to a reduction in the preparation time [24]. Sarkar *et al*. (2014) have examined a polymeric flocculant based on amylopectin grafted with poly (acrylic acid) (g-AP) for the treatment of a synthetic effluent as well as various industrial wastewaters. The results obtained showed that g-AP could be used as an effective flocculant for removing the suspended silica particles as well as the cationic and anionic colors of aqueous solutions under different pH conditions [25]. Liu *et al*. (2014) have examined the effect of an effective eco-friendly cellulose-based flocculant, BPC-gPAM, for the treatment of effluent from paper mill waste. The

results obtained showed that in neutral and acidic conditions, the BPC-gPAM flocculant had a better efficiency than polyacrylamide for the treatment of plant wastewater. The BPC-gPAM flocculant is a suitable option for usage in industries due to its biodegradability and low cost, and removal of cadmium particles from paper plant waste [26]. Wang *et al*. (2014) have studied the characterization of the dewatering process of activated sludge assisted by cationic surfactants. During the experiments, it was found that surfactant cetyl trimethyl ammonium bromide (CTAB) was more effective than the surfactant dodecyl trimethyl ammonium bromide (DTAB) for the release of water bonded to solid particles. The surfactant significantly increased the dewatering efficiency of the pulp due to reduction in the resistance and moisture [27]. Lihong *et al*. (2011) have studied the enhancement of the efficiency of the filtration process with filter aids such as diatomaceous earth and wood pulp cellulose. Investigations showed that by adding filter aids, the filtration rate increased and the moisture content of cake decreased due to changes in the structure, porosity, compressibility, hydraulic resistance, and cake permeability [28]. Wang *et al*. (2009) have examined the effect of a novel flocculant with a high water-solubility (Chitosan-g-PDMC) on the factory effluent. The studies have shown that Chitosan-g-PDMC has an excellent flocculation capacity and that the flocculation efficiency is much better than polyacrylamide [29]. In another research work, Dias *et al*. (2003) have examined the effect of adding the chemicals to the vacuum filtration of ultrafine hematite iron concentrate. In this work, a type of flocculant with the commercial name Flonex 9076 with a high molecular weight and two types of surfactants, CP-00DA038/Nalco and Aerodri 100/Cytec, were used. The results obtained showed that with increase in the amount of flocculants, the specific cake resistance and cake formation time were reduced, and the cake porosity and cake moisture increased. Also the interaction between the surfactant and the flocculant was also studied, in which case the moisture content was reduced to a small value [30].

The aim of this work was to investigate the effect of different additives including flocculant, coagulant, surfactant, and filter aid on the filtration responses such as the cake formation rate, cake moisture content, specific cake resistance, resistance of filter cloth, and recovered water content. The vacuum top-feed method was applied for dewatering the copper concentrate. In this work, for the first time, the effect of interaction of the effective additives as chemical reagents on the filtration process was investigated. In this research work, the effects of flocculants (5, 15, and 25 g/t), dissolving filter aids (100, 250, and 400 g/t), solid filter aids (1, 2.5, and 5 wt.%), surfactants (3, 9, and 15 cc/l), and coagulants (0.5, 1.5, and 2.5 g/t) on the filtration efficiency were precisely investigated with the two methodologies OFAT (one-factor-at-a-time) and the statistical analysis. In the OFAT methodology, each additive was optimized separately. Also statistical analysis was done using the DX7 software and the Central Composite Design (CCD) method. The statistical analysis was used to evaluate the efficiency of the simultaneous usage of the influential additives in the filtration process, and consequently, find the optimal condition with the maximum cake formation rate and the minimum cake moisture content.

## 2. Material and methods
### 2.1. Samples
The sample used for the filtration experiments was originated from the Qaleh Zari copper mine. The sampling was performed at one hour intervals after the beginning of each shift and during 20 days from the concentrate thickener underflow (filter input). At the end of each work day, the samples were transferred to the mineral processing laboratory of the Qaleh Zari copper complex, and were packed and dried after calculating their solid percent. The Qaleh Zari copper concentrator plant has a 400-ton capacity per day, and its feed is entered into the flotation cells after two stages of grinding (rod and ball mills). The concentrate of the flotation process is entered into the concentrate thickener for dewatering and is then dried by a filter. The optimal performance of the filtration process is led to make a dried and ready-to-sale concentrate and with returning of the recycled water to the processing circuit. It will be of great help to solve the water shortage challenge in a semi-arid area. The final dewatering is done in the circuit using a vacuum drum filter. The volume of the filter pond is $4m^3$ and the capacity of 40-45 ton/day of copper concentrate with a cake moisture content of 15%.

### 2.2. Characterization study
The semi-quantitative X-ray diffraction (SQXRD) technique was used to define the main and trace minerals in the representative sample. The X-ray powder diffraction patterns were obtained using a PHILIPS PW1800 diffractometer with Ni-filtered Cu–Ka radiation, and a goniometer speed of 1o $2\theta$/min. The diffraction profiles with a 0.01 precision of d-spacing measurements were

conducted from 4° to 60° (2θ). Based on the XRD results, chalcopyrite (CuFeS$_2$ = 30%), pyrite (FeS$_2$ = 23%), quartz (SiO$_2$ = 12%), and galena (PbS = 10%) were distinguished as the major phases of the sample, and sphalerite (ZnS) and hematite (Fe$_2$O$_3$) were the minor phases. These minerals were reported in an abundant order in the XRD graph (Figure 2).
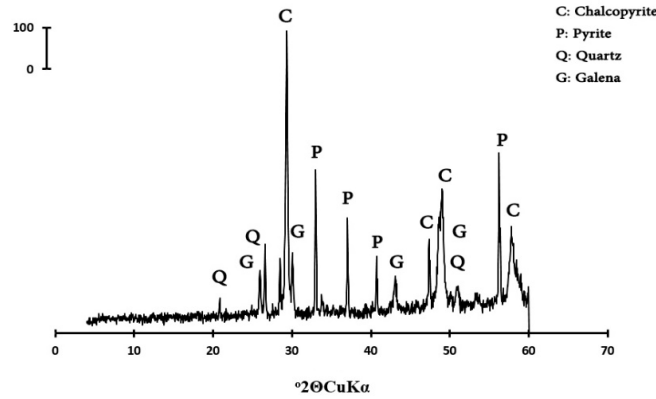


**Figure 2. XRD of the concentrate sample prepared from the Qaleh Zari copper mine.**

The representative sample was also analyzed using the X-ray fluorescence (XRF) technique in order to determine the major and minor elements and oxides. The result of XRF analysis (PHILIPS PW1480) showed that Fe, S, SiO$_2$, Cu, and Al$_2$O$_3$ were the major components, and other oxides such as CaO, K$_2$O, MgO, Na$_2$O, P$_2$O$_5$, and TiO$_2$ were the minor components presented in the concentrate sample. The XRF analysis results are presented in Table 1. More than 75% of the original sample was formed from Fe, S, and Cu. It is noteworthy that the Qaleh Zari's sulfide sample contains gold and silver, and the process works with a maximum recovery and a minimum copper grade.

**Table 1. XRF analysis of the concentrate sample prepared from the Qaleh Zari copper mine.**

| | Component | Fe | S | SiO$_2$ | Cu | Al$_2$O$_3$ | |
|---|---|---|---|---|---|---|---|
| Major | % | 38.51 | 28.52 | 18.67 | 10.65 | 1.97 | |
| | Component | CaO | K$_2$O | MgO | Na$_2$O | P$_2$O$_5$ | TiO$_2$ |
| Minor | % | 0.42 | 0.31 | 0.24 | 0.05 | 0.038 | 0.071 |
| | Component | L.O.I | | | Total | | |
| | % | 0.02 | | | 99.469 | | |

The density measurement was performed twice by a pycnometer with a representative sample. Due to the presence of frother in the sample and making mistake during the identification of the density by water, kerosene was used instead of water in one of the experiments. After performing the experiment with water, the density was measured to be 4.02 $g/cm^3$, and after using kerosene, the density was 4.025 $g/cm^3$. In general, a density of 4.03 $g/cm^3$ was used for the next step of filtration tests. The density calculation was performed using Equation 5.

$$\rho_s = \frac{(P_s - P_o)}{(P_w - P_o) - (P_p - P_s)} * \rho_w \qquad (5)$$

where $P_o$ is the weight of the empty pycnometer; $P_s$ is the weight of the pycnometer and solid; $P_o$ is the weight of pycnometer, water, and solid; $\rho_w$ is the weight of pycnometer and water; $\rho_w$ is the density of water, and $\rho_s$ is the solid density.

Due to the presence of materials of small particle size and also for a higher accuracy, the wet screening analysis method was used for determining the size distribution of the sample whose result is demonstrated in Figure 3. As a result, d$_{50}$, d$_{80}$, and d$_{90}$ were determined to be 66.6, 120.3, and 139.5 microns, respectively.
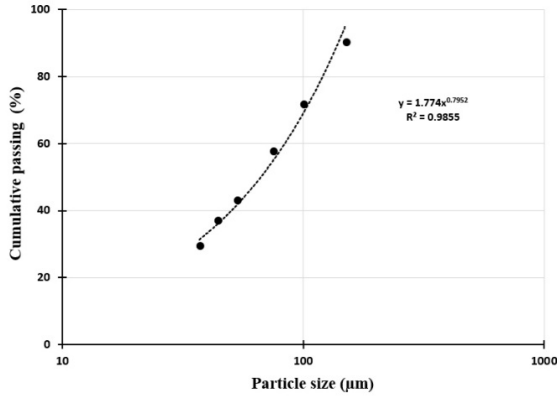
**Figure 3. Particle size distribution of the representative sample of copper concentrate.**



**Figure 4. Determination of ZPC of copper concentrate by the MT method.**

The Zero point of charge (ZPC) of minerals is determined in two ways: using a device and using the mass titration (MT) method. In this research work, the surface charge of particles was measured by mass titration. The procedure for the mass titration method is as follows [31]:

1. Preparation of 50 mL of 0.01 M NaCl solution;
2. To remove the existing $CO_2$, boil the solution and then read the pH of the solution;
3. pH adjustment at pH = 2 using HCl or NaOH;
4. Add 150 mg of the material to solution and mix the solution with 1500 rpm for 48 to 72 hours at the ambient temperature;
5. Repeat the above steps at pH values of 4, 6, 8, 10, and 12;
6. Filtering the solution after the determined time and pH reading;
7. Draw the primary pH (x) graph in terms of the final pH read (y) and draw the line y = x.

By following the above steps and plotting the graph and line y = x, the upper part of the line, the negative section, and the bottom part of the line will be positive. The intersection of the line y = x with the plotted graph shows $pH_{zpc}$. Figure 4 shows the ZPC graph by the MT method. According to Figure 4, ZPC is achieved within a neutral pH range.
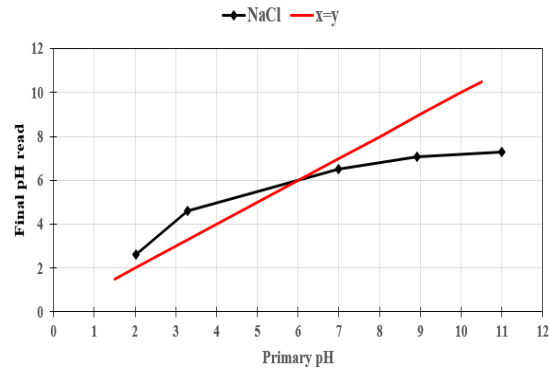
## 2.3. Reagents

In this research work, different materials such as flocculant, surfactant, coagulant, dissolving filter aid, and solid filter aid were used in order to evaluate the filtration experiments. Flocculants are organic polymers with long molecular chains that are dissolved in water. Flocculation is a process that involves the formation of aggregates, and settles down the colloidal particles [25]. Flocculation, which is widely applied because of its high efficiency and facile operation, is one of the most important industrial processes for water treatment. Selection of the flocculant type has a direct impact on the water recycling process [32]. Two of the most widely types of flocculants used in the industry are inorganic metal-based flocculants and synthetic polymers [33]. In terms of the chemical structure (Figure 5), flocculants are also classified into the three categories of anionic flocculants (carboxymethyl cellulose and polyacrylates), cationic flocculants (polyethyleneamine and polyvinylamine), and non-ionic flocculants (polysaccharides, dextrin, and polyacrylamides) [34, 35].
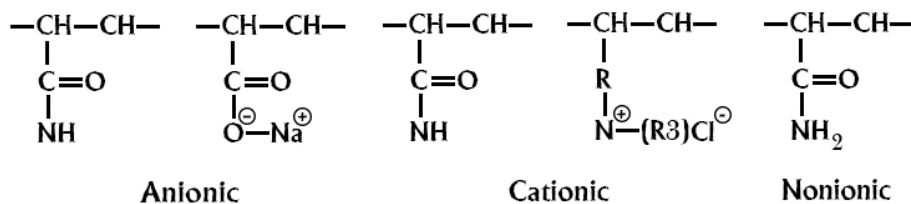


**Figure 5. Flocculant types and their chemical formula [34].**

In the flocculation process, the optimal amount of flocculant must be determined because a less flocculant usage does not cause flocculation. In addition, using more than the optimal dosage causes a strict stabilization phenomenon [35]. The characteristics of the flocculants used in this work

are presented in Table 2. The effect of each one of the above-mentioned flocculants with concentrations of 5, 15, and 25 g/t on the filtration process was thoroughly investigated.

**Table 2. Characteristics of the flocculants used in the tests.**

| Characteristic | A25 | A26 | A28 |
|---|---|---|---|
| Physical state | Solid | Solid | Solid |
| Appearance | Colorless or white granular powder | Colorless or white granular powder | Colorless or white granular powder |
| Odor | None | None | None |
| Average molecular weight, g/mol | 12000-15000 thousands | 14000-18000 thousands | 18000-20000 thousands |
| Color | white | white | white |
| Particle size | 20-80 mesh | 20-80 mesh | 20-80 mesh |
| Charge | Anionic | Anionic | Anionic |
| Degree of hydrolysis | Low | Medium | Medium |
| Solubility in water | Soluble | Soluble | Soluble |

In the coagulation process, the surface charge of particles is neutralized by the addition of chemicals with opposing electrical charges, which results in the accumulation of particles. To neutralize the negative charge on particles, mineral salts such as lime, iron sulfate, and aluminum sulfate (which include different cations such as $Ca^{2+}$, $Fe^{3+}$, and $Al^{3+}$) are used. To neutralize the positive charge, phosphates (sodium metaphosphate or calgon) are used. In this work, $BaCl_2$, $CaCl_2 . 2H_2O$ and $KCl$ were used as the coagulants in concentrations of 0.5, 1.5, and 2.5 g/t. The characteristics of the coagulants used in the experiments are presented in Table 3.

**Table 3. Specifications of the coagulants used in the experiments.**

| Coagulant | Anion charge capacity | Cationic charge capacity |
|---|---|---|
| $CaCl_2 . 2H_2O$ | 1 | 2 |
| $BaCl_2$ | 1 | 2 |
| $KCl$ | 1 | 1 |

Filter aids are materials that, by forming a porous layer on the filter media, improve the flow of water from the media. Dissolving filter aids are non-polymeric compounds and include one or more hydrophiles (sulfonate, carboxylate, ethoxylate) and hydrophobic groups, and usually have a long chain of hydrocarbons [36, 37]. Pulp filtration including bentonite clay, kaolin fine grains, and other gelatinous components is very difficult. For this reason, these pulps are mixed with solid filter aids. These filter aids are added in a powder form or by a certain size distribution. Solid filter aids with changes in structure, porosity, compressibility, hydraulic resistance, and cake permeability increase the filtration rate and decrease the cake moisture content. This material improves the efficiency of filtration due to its high area per unit weight. It is noteworthy that these substances added to the environment must be non-reactive [38]. Generally, perlite, diatomite, and cellulose are the best solid filter aids that are used by the industries. In this work, solid filter aids were used in the amounts of 1, 2.5, and 5 wt. %, and dissolving filter aids were used at concentrations of 100, 250, and 400 g/t.

## 2.4. Filtration experiments and operational apparatus

The vacuum filtration experiment was performed via the vacuum top-feed leaf method using a Buchner funnel, a graduated cylinder, and a vacuum manufacturing system (Figure 6). The purpose of performing the experiment with this method was to evaluate the filtration responses such as the filtration rate, specific cake resistant, cake compression, and filtration interface resistance. The samples with the weight of 350 g were used to perform the filtration experiments. At the end of each test, the moist cake was placed in a dryer for 24 h at a temperature of 75 ℃. The filtrate volume was measured for 10 min; in this way, in the first 5 min, the intervals were considered to be 10 s, and then the intervals were 50 s.
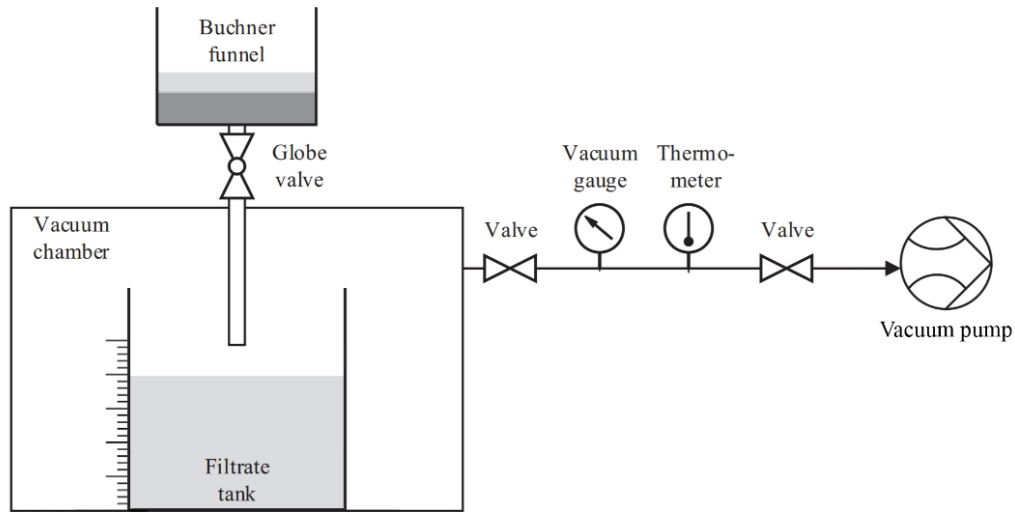
**Figure 6. Operational apparatus of filtration process in lab.**

In this work, the effects of flocculants (5, 15, and 25 g/t), dissolving filter aids (100, 250, and 400 g/t), solid filter aids (1, 2.5, and 5 wt.%), surfactants (3, 9, and 15 cc/l), and coagulants (0.5, 1.5, and 2.5 g/t) on the filtration efficiency were precisely investigated by the two methodologies of OFAT (one-factor-at-a-time) and statistical analysis. In evaluating the results obtained, the filtration responses of the cake formation rate, cake moisture content, resistance to filter media (R), specific cake resistance (α), and water recovery were considered. In the OFAT methodology, each additive was optimized separately. The additives and their qualitative and quantitative levels are presented in Table 4.

**Table 4. Additives and their tested levels by the OFAT methods.**

| Parameter | Type | Number of levels | Dosage |
|---|---|---|---|
| Flocculants | Qualitative | 3 | |
| Flocculants | Quantitative | 3 | 5, 15 and 25 g/t |
| Dissolving filter aids | Qualitative | 2 | |
| Dissolving filter aids | Quantitative | 3 | 100, 250 and 400 g/t |
| Solid filter aids | Qualitative | 2 | |
| Solid filter aids | Quantitative | 3 | 1, 2.5 and 5 wt. % |
| Surfactants | Qualitative | 3 | |
| Surfactants | Quantitative | 3 | 3, 9 and 15 cc/l |
| Coagulants | Qualitative | 3 | |
| Coagulants | Quantitative | 3 | 0.5, 1.5 and 2.5 g/t |

The statistical analysis was done using the DX7 software via the Central Composite Design (CCD) method. The types of additives and their dosages analyzed by the CCD method are presented in Table 5. For evaluating the effects of the parameters presented in Table 5, the cake formation rate (mm/s) and moisture content (%) were considered as the responses. The statistical analysis was used to evaluate the efficiency of the simultaneous usage of three influential additives in the filtration process, and consequently, to find the optimal condition with the maximum cake formation rate and minimum moisture content.

**Table 5. Types of additives and their dosages analyzed by the CCD method.**

| Additive | Code | Unit | Lowest level (-1) | Middle level (0) | Highest level (+1) |
|---|---|---|---|---|---|
| Flocculant (A25) | $X_1$ | g/t | 2.91 | 13 | 23.09 |
| Solid filter aid (perlite) | $X_2$ | wt.% | 0.31 | 2.5 | 4.69 |
| Surfactant (Cop20-101) | $X_3$ | cc/l | 0.48 | 3 | 5.52 |

For the statistical tests, 600 s was considered as the filtration time. The polyester filter cloth with a twill texture was used as the filtration medium. Figure 7 shows the microscopic image of the filter cloth taken by a binocular microscope (True Chrome

matrix model). During the preliminary experiments, the optimal amounts of solid percent, pH value for the pulp, and pressure differences were 55%, ambient pH, and 0.7 bar, respectively.
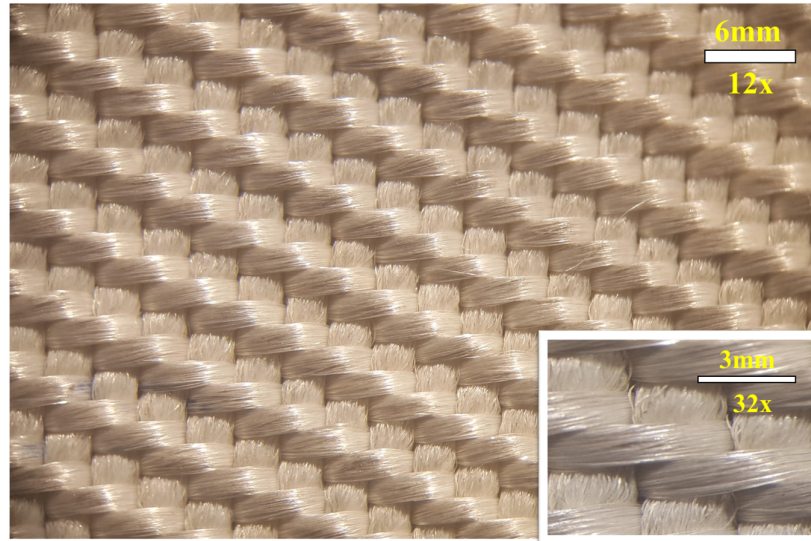


**Figure 7. Microscopic image of the filter cloth used in the experiments.**

## 3. Results and discussion
### 3.1. OFAT methodology
#### 3.1.1. Anionic flocculants

Since the pH value for the pulp was close to ZPC, both the anionic and cationic flocculants were used in the experiments. Flocculants, by binding solid particles together, cause larger clots to form. As shown in Figures 8A, 8B, and 8C, by increasing the concentration of the flocculent and by binding the small solid particles present in the pulp to each other and the formation of larger clots, the specific cake resistance increased, which could be attributed to the smoothness of the pulp particles. For this reason, since the flocculant concentration increased, the resistance to filter cloth decreased. The highest amount of the specific cake resistance with a value of 3.3 kg/m × 10$^{12}$ is related to the concentration of 5 g/t of the flocculant A26 (Figure 8B), and the minimum specific cake resistance with a value of 1.08 kg/m × 10$^{12}$ is related to the concentration of 25 g/t for the flocculant A25.

By increasing the concentration of the flocculant, the size of the particles formed also increases. This increase in size will cause the water in the pulp to flow out at a faster rate when force is applied. Increasing the particles size, on the other hand, causes water to be trapped between the particles,

which has a negative effect on the moisture content of the filter cake and increases it. Figures 8D, 8E, and 8F show the effect of increasing the concentration of the flocculant on the cake formation rate and cake moisture content. According to the results presented in the above-mentioned figures, an increase in the flocculant concentration leads to an increase in the cake formation rate and cake moisture content. According to Figure 8E, with increase in the concentration of the flocculant A26, the cake formation rate and moisture content increased but it is noteworthy that by increasing the concentration of the flocculant A26 from 15 g/t to 25 g/t, the rate of cake formation did not increase significantly and underwent an almost constant process. Due to fixing the cake formation rate at concentrations greater than 15 g/t, it can be concluded that increasing the concentration of the flocculant A26 does not increase the formation of clots. Due to the uniformity of the clots formed at 25 g/t, the moisture content of the filter cake was not significantly increased compared to the concentration of 15 g/t, and only 0.06 % was increased. The highest cake formation rate was obtained at a concentration of 25 g/t for the flocculant A25 (Figure 8D).
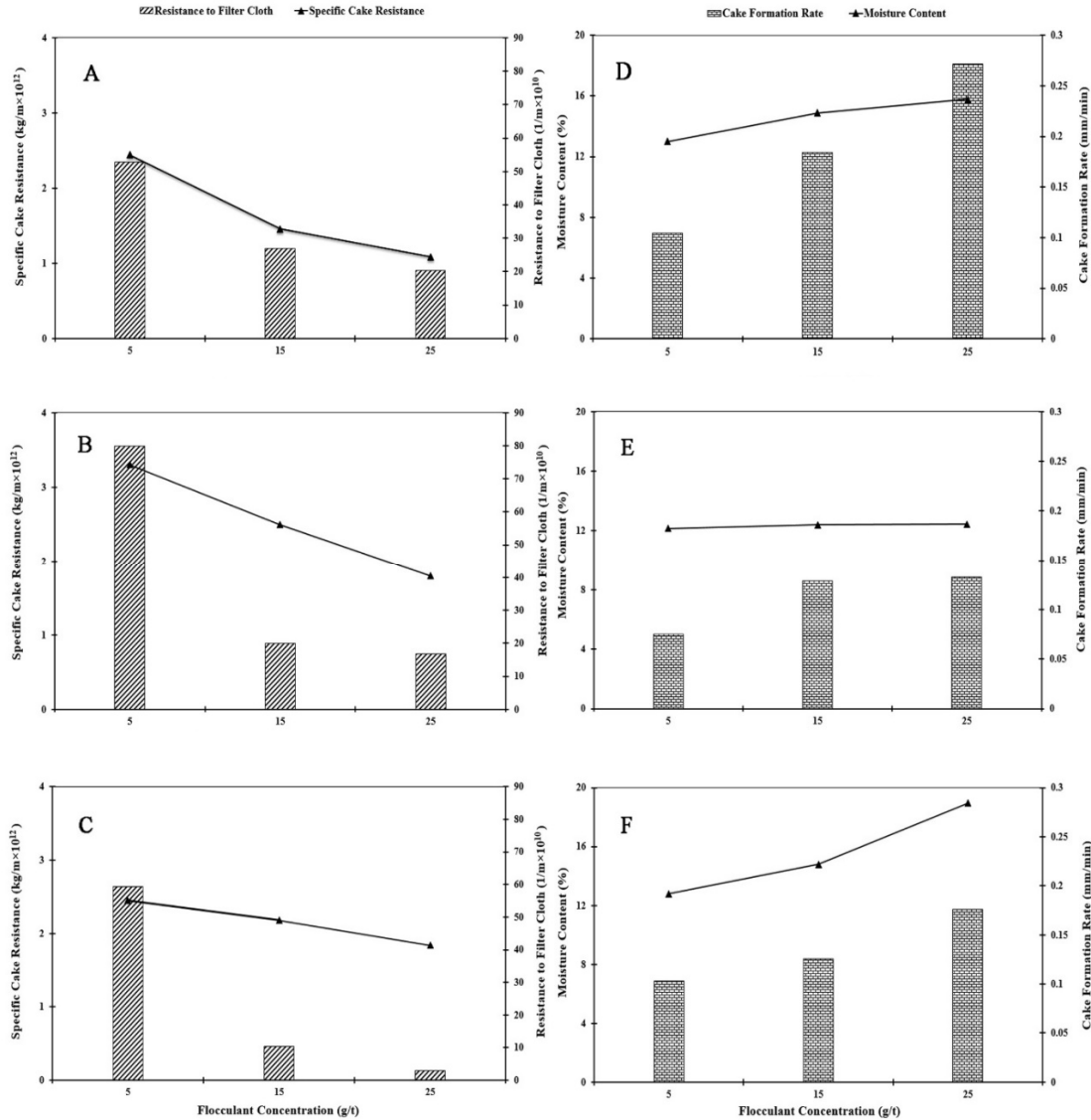
**Figure 8. Effect of anionic flocculants on the specific cake resistance, resistance to filter cloth, moisture content, and cake formation rate, A25 (A and D), A26 (B and E), and A28 (C and F).**

### 3.1.2. Cationic flocculants

Increasing the concentration of the flocculant, which also increases the density and viscosity of the pulp, sometimes causes problems such as increasing the resistance to filter cloth and reducing the capacity of the vacuum generator. On the other hand, a high concentration of the flocculant also increases its preparation time. By considering the pH of the pulp and ZPC of the sample, the role of cationic flocculants is expected to be less than that of the anionic ones.

According to Figure 9A, the specific cake resistance was reduced by increasing the concentration of the flocculant C25, which was more obvious at a concentration of 25 g/t. The

resistance to filter cloth was also expected to reduce by increasing the concentration of the flocculant but it increased at a concentration of 15 g/t. The reason for this phenomenon, as noted earlier, can be the high molecular weight of the cationic flocculants compared to the anionic ones. Increasing the molecular weight of the flocculant increases the viscosity of the pulp, making it difficult to pass through the fluid from the cloth pores. The effect of the flocculant C26 on the specific cake resistance and the resistance to filter cloth is presented in Figure 9B. Based on the results presented in this figure, the flocculant C26 played its role in reducing the amount of the specific cake resistance. However, after the

concentration of 15 g/t for this flocculant, the resistance to filter cloth did not decrease and it underwent a constant process. The same trend happened for the flocculant C28 (Figure 9C). The reason for the decrease in the amount of specific cake resistance at high concentrations of the flocculants C26 and C28 can be attributed to the formation of larger clots. The effect of cationic flocculants on the cake formation rate and cake moisture content are presented in Figures 9D, 9E, and 9F. The highest cake formation rate with a value of 0.163 mm/s is related to the concentration of 25 g/t for the flocculant C26 (Figure 9E).

Table 6 shows the results of the optimal condition of each flocculant in the filtration process. The flocculants C25 and C28 are not suitable due to fluctuations in the resistance to filter cloth by increasing the concentration of the flocculant and also the higher moisture of the filter cake at higher concentrations. The graph of water recovery versus time for the four flocculants A25, A26, A28, and C26 is presented in Figure 10. Based on the results obtained, the flocculant A25 with a concentration of 15 g/t was selected as the optimal one due to the minimum specific cake resistance and filtration duration as well as the maximum water recovery rate and cake formation rate.
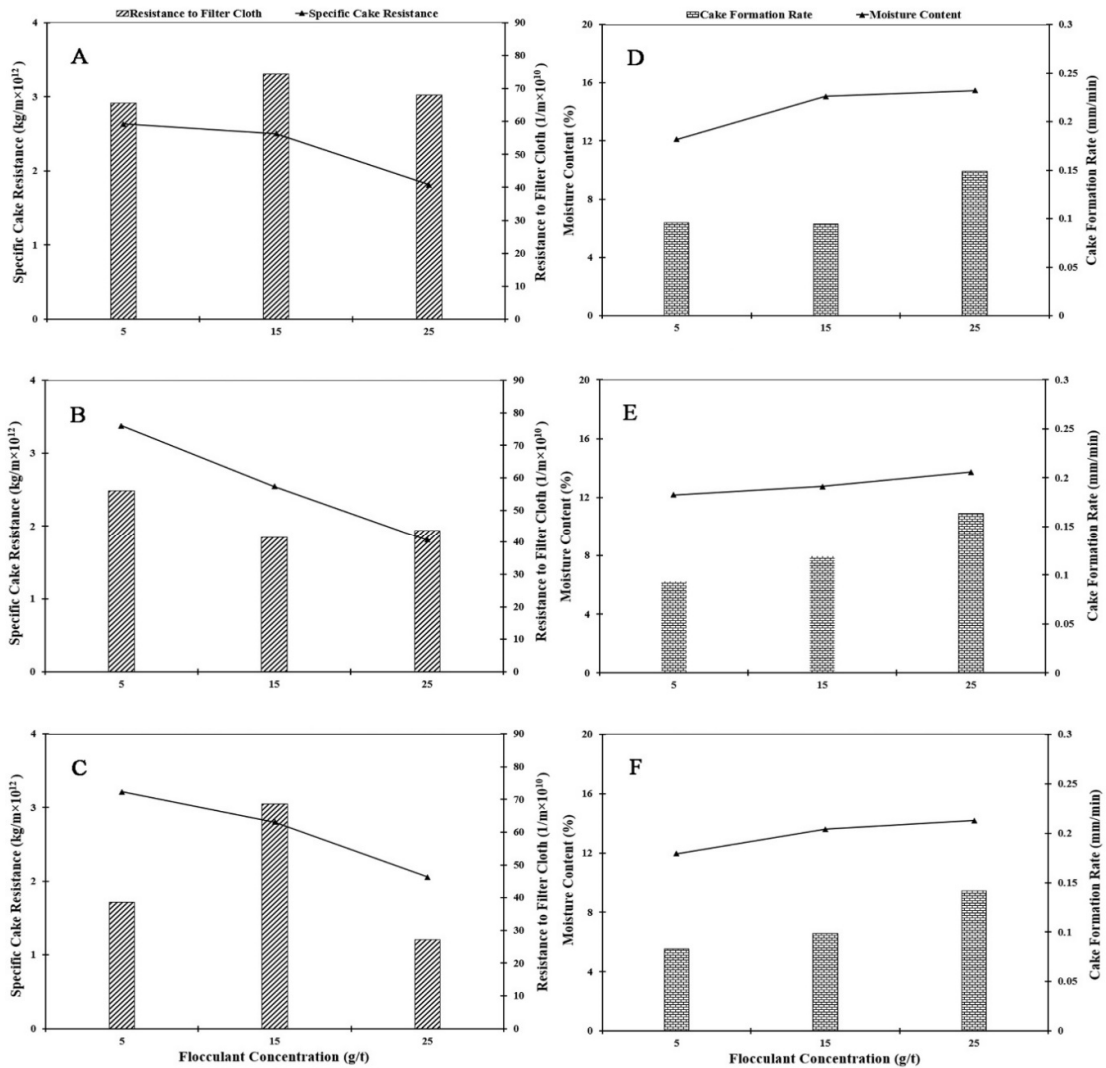


**Figure 9. Effect of cationic flocculants on the specific cake resistance, resistance to filter cloth, moisture content, and cake formation rate, C25 (A and D), C26 (B and E), and C28 (C and F).**
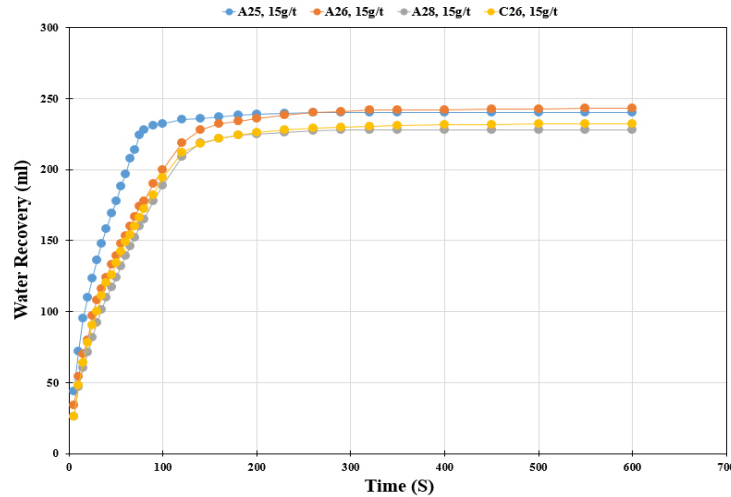
**Figure 10. Effects of the flocculants A25, A26, A28, and C26 on the water recovery.**

**Table 6. Effects of different anionic and cationic flocculants on the filtration process.**

| Flocculant | Optimal concentration (g/t) | Cake formation rate (mm/s) | Moisture content (%) | Specific cake resistance (kg/m$\times 10^{12}$) | Resistance to filter cloth ($m^{-1} \times 10^{10}$) | Filtration time (s) | Pressure at the end of dewatering (bar) |
|---|---|---|---|---|---|---|---|
| A25 | 15 | 0.184 | 14.88 | 1.46 | 26.94 | 260 | 0.4 |
| A26 | 15 | 0.129 | 12.41 | 2.49 | 19.89 | 550 | 0.6 |
| A28 | 15 | 0.126 | 14.80 | 2.18 | 10.44 | 290 | 0.4 |
| C25 | 5 | 0.096 | 12.12 | 2.63 | 65.48 | 550 | 0.4 |
| C26 | 15 | 0.120 | 12.74 | 2.55 | 41.57 | 500 | 0.4 |
| C28 | 5 | 0.083 | 11.96 | 3.21 | 38.63 | 550 | 0.4 |

### 3.1.3. Filter aids

The filter aid reduces the compressibility and increases the porosity of the cake, which leads to a decrease in the specific cake resistance and an increase in the cake formation rate and improving the dewatering condition. At higher concentrations of the filter aid in the liquid phase, due to the effect of its molecular weight, the increase in the viscosity of the pulp, the drop in the cake formation rate, and the increase in the moisture content of the filter cake were observed. Obviously, a higher viscosity is created by the filter aid with a higher molecular weight. Usually, in the industry, due to the economic justification and the availability of perlite and diatomite, these two materials are used as the solid filter aid. Figure 11 shows the effect of the filter aid on the filtration process. Due to the high density of the aero100 and aero104 as the liquid filter aids, increasing the concentration of these filter aids negatively affects the cake formation rate and reduces it (Figures 11A and 11B). Due to the higher density of the aero104 filter aid rather than the aero100 one, increasing the concentration of this filter aid has a more influential effect on reducing the cake formation

rate. Figures 11C and 11D show the influence of perlite and diatomite as solid filter aids on the filtration process. According to Figure 11C, by increasing the perlite content from 1% to 5%, the cake formation rate and cake moisture content increase. The cake formation rate increased from 0.083 mm/s to 0.273 mm/s, and the moisture content of the filter cake increased from 12.36% to 12.98%. Based on the maximum cake formation rate in the amount of 5% perlite, this dosage of perlite filter aid was selected as the optimal one. However, due to the low density of perlite, providing 5% of this filter aid is not practically feasible or affordable, so the amount of 2.5% was considered as the optimal value. According to Figure 11D, an increase in the amount of the diatomite filter aid had a positive effect on the cake formation rate, and it increased from 0.069 mm/s in the amount of 1% to 0.133 mm/s in the amount of 5%. The upward trend of the cake formation rate is related to the increase in porosity (explained previously). Table 7 is provided for comparing the filter aids and selecting the best one, and shows the effect of the optimal concentration of each filter aid on the filtration process. According to the results

presented in this table, the perlite with an amount of 2.5% was selected as the optimal value of filter aid, due to the maximum values of the cake formation rate and the pressure difference at the

end of the dewatering as well as the minimum operational parameters such as the duration of the filtration and the specific cake resistance, and the desired amount of moisture content of the cake.
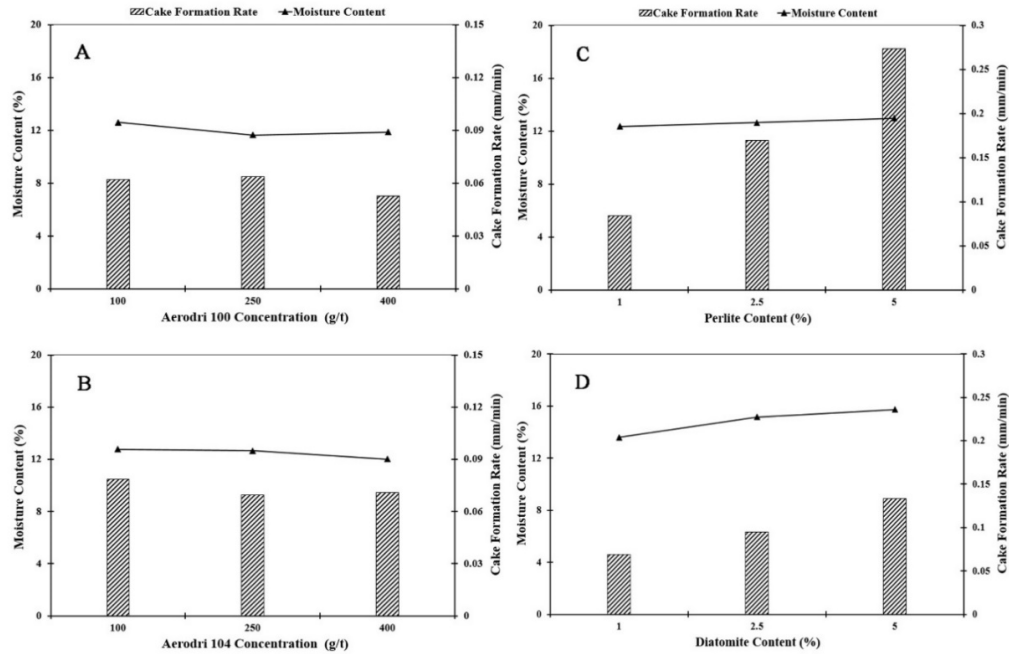


**Figure 11. Effects of aero100 (A), aero104 (B), perlite (C), and diatomite (D) as filter aids on the cake formation rate and moisture content.**

**Table 7. Effects of filter aids on the filtration process.**

| Filter aid | Optimum amount | Cake formation rate (mm/s) | Moisture content (%) | Specific cake resistance $(kg/m \times 10^{12})$ | Resistance to filter cloth $(m^{-1} \times 10^{10})$ | Filtration time (s) | Pressure at the end of dewatering (bar) |
|---|---|---|---|---|---|---|---|
| Aerodri100 | 250 (g/t) | 0.064 | 11.65 | 3.76 | 61.12 | 550 | 0.62 |
| Aerodri104 | 100 (g/t) | 0.078 | 12.76 | 4.31 | 68.75 | 500 | 0.58 |
| Perlite | 2.5 (%) | 0.170 | 12.65 | 1.82 | 88.41 | 450 | 0.70 |
| Diatomite | 1 (%) | 0.069 | 13.59 | 4.10 | 62.42 | 500 | 0.62 |

### 3.1.4. Surfactants

At this stage, the three surfactants Cop2584, Cop20-101, and AID were used to evaluate the filtration efficiency. Absorption of the surfactant in the gas-liquid interface reduces the surface tension of the liquid and also increases the hydrophobicity of the solid particles. Increasing the concentration of surfactants, and consequently, increasing the viscosity of the pulp has a negative effect on the specific cake resistance and also the cake formation rate.

Figure 12A shows the effect of the surfactant Cop2584 on the two responses of cake formation rate and cake moisture content. The results obtained showed that with increase in the concentration of surfactant, the moisture content of the cake reached from 12.75% to 12.68% with a

decreasing trend. On the other hand, by increasing the concentration of Cop2584 from 3 cc/l to 15 cc/l, the cake formation rate increased so that in the concentration of 15 cc/l, it reached the maximum value of 0.068 mm/s. The effect of the surfactant Cop2584 on the specific cake resistance and the resistance to filter cloth is demonstrated in Figure 12B. Based on the results presented in this figure, the maximum resistance to filter cloth and the specific cake resistance were obtained at a concentration of 3 cc/l. Also the resistance to filter cloth and the specific cake resistance did not have a significant difference in the concentrations of 3 cc/l and 15 cc/l. By examining the results obtained, it was found that the addition of the surfactant Cop2584 had no significant effect on the efficiency of the filtration process but a concentration of 3 cc/l

for the surfactant Cop2584 had a better condition than the other concentrations. As shown in Figure 12C, an increase in the concentration of the surfactant Cop20-101 caused a significant decrease in the cake formation rate from 0.12 mm/s to 0.066 mm/s, which was also predictable due to the increase in the specific cake resistance for increasing the concentration of the surfactant (Figure 12D). Figure 12E shows the effect of the surfactant AID on the cake formation rate and cake moisture content. As shown in this figure, by increasing the concentrations, the cake formation rate was also increased and reached the highest value at a concentration of 15 cc/l, which was equal to 0.178 mm/s. It should be noted that in the concentration of 15 cc/l, the specific cake resistance is also at its lowest value (Figure 12F). By increasing the concentration of the surfactant AID, the moisture content increases. The optimal concentrations of the surfactants Cop2584, Cop20-101, and AID and their effects on the filtration process are presented in Table 8. According to the results obtained, the highest cake formation rate and the lowest filtration time was related to the surfactant AID with a concentration of 15 cc/l. On the other hand, the minimum moisture content of the cake, the specific cake resistance, and the resistance to filter cloth were obtained using the surfactant Cop20-101 with a concentration of 3 cc/l. Figure 13 shows the water recovery versus time for the three surfactants AID, Cop2584, and Cop20-101 with the concentrations of 15, 3, and 3 cc/l, respectively. As shown in this figure, the best dewatering rate is related to the surfactant Cop20-101 at a concentration of 3 cc/l, and the lowest dewatering rate is related to the surfactant AID at a concentration of 15 cc/l. Based on the results obtained, the surfactant Cop20-101 with a concentration of 3 cc/l was selected as the optimal condition due to the lowest cake moisture content, specific cake resistance, and resistance to filter cloth and also an acceptable cake formation rate.
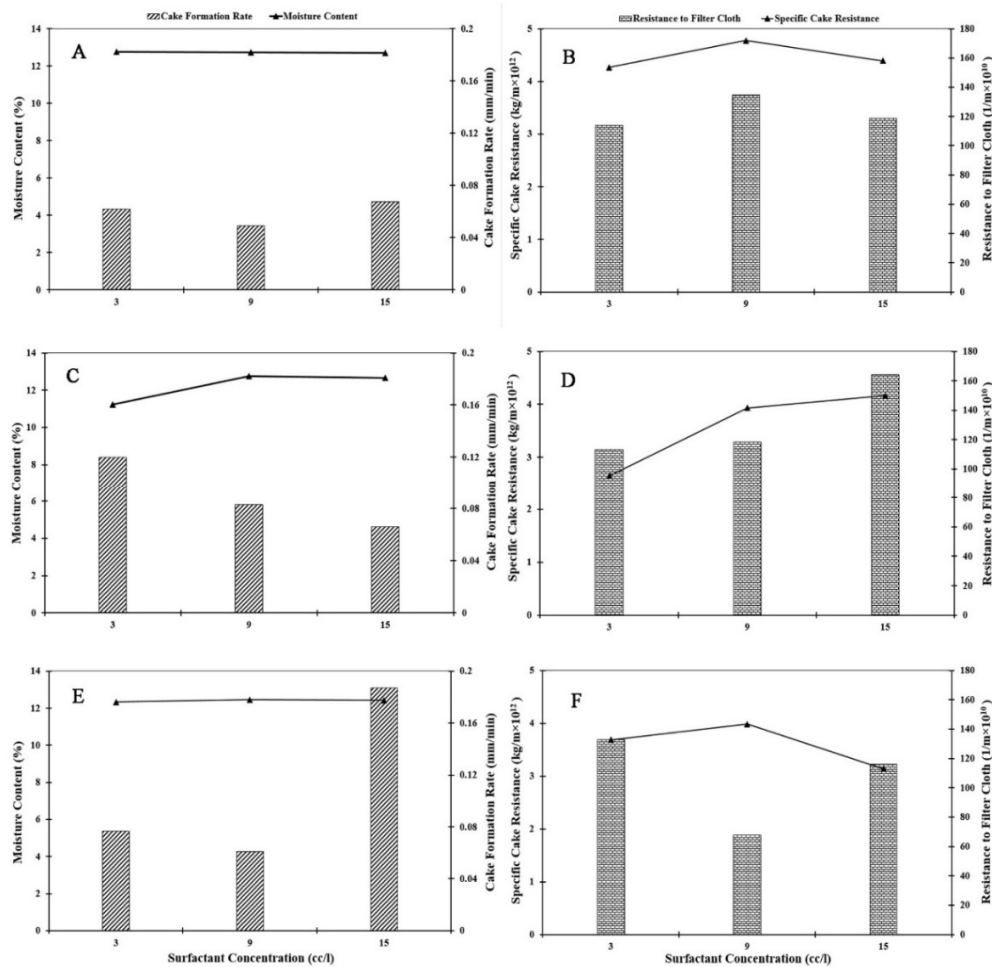


**Figure 12. Effects of different surfactants on the moisture content, cake formation rate, specific cake resistance, and resistance to filter cloth, Cop2584 (A and B), Cop20-101 (C and D), and AID (E and F).**
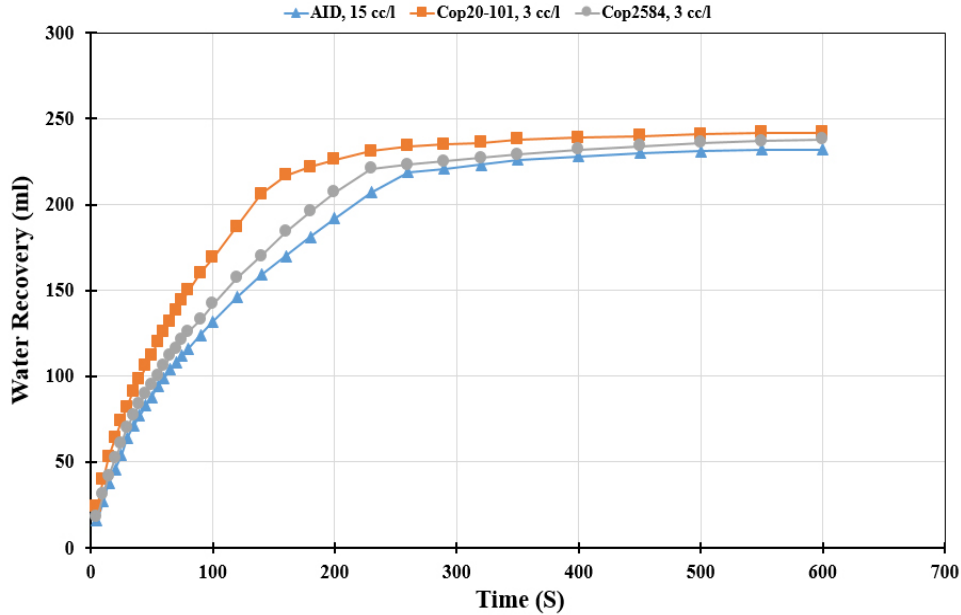
**Figure 13. Effects of the surfactants Cop2584, Cop20-101, and AID on the water recovery.**

**Table 8. Effects of the surfactants on the filtration process.**

| Surfactant | Optimal concentration (cc/l) | Cake formation rate (mm/s) | Moisture content (%) | Specific cake resistance (kg/m×$10^{12}$) | Resistance to filter cloth ($m^{-1} \times 10^{10}$) | Filtration time (s) | Pressure at the end of dewatering (bar) |
|---|---|---|---|---|---|---|---|
| AID | 15 | 0.187 | 12.33 | 3.14 | 116.01 | 550 | 0.7 |
| Cop2584 | 3 | 0.062 | 12.57 | 4.27 | 113.99 | 600 | 0.7 |
| Cop20-101 | 3 | 0.120 | 11.22 | 2.65 | 113.21 | 600 | 0.7 |

### 3.1.5. Coagulants

The effects of the three coagulants $CaCl_2.2H_2O$, $KCl$, and $BaCl_2$ on the filtration process were investigated. As mentioned earlier, coagulants improve the dewatering process by neutralizing the particle surface charge. The effect of *the* coagulant $CaCl_2.2H_2O$ on the cake formation rate and cake moisture content is presented in Figure 14A. The process of changing the cake formation rate was initially increased by increasing the concentration of the coagulant to 1.5 g/l. However, with an increase in the concentration of the coagulant $CaCl_2.2H_2O$ up to 2.5 g/l, no change was observed in the cake formation rate. Also the coagulant $CaCl_2.2H_2O$ had no significant effect on the moisture content of the cake so that with increase in the coagulant concentration, the moisture content was increased only by 0.3 %. The coagulant KCl, like $CaCl_2.2H_2O$, had no effect on the moisture content so that the difference between the maximum and minimum moisture contents was only 0.1% (Figure 14B). According to this figure,

the highest amount of cake formation rate was found to be 0.057 mm/s at a concentration of 1.5 g/l for the coagulant KCl. Figure 14C shows the effect of the coagulant $BaCl_2$ on the cake formation rate and cake moisture content. Based on the results obtained, the coagulant $BaCl_2$ did not have a significant effect on the cake formation rate so that the highest and lowest difference in the cake formation rates was only 0.01 mm/s. The minimum and maximum moisture contents of the cake were 12.3% and 16.3%, respectively. The optimal concentrations of the coagulants $CaCl_2.2H_2O$, $KCl$, and $BaCl_2$, and their effects on the filtration process are presented in Table 9. The highest cake formation rate, the lowest amount of resistance to filter cloth, and also the lowest amount of specific cake resistance are related to the coagulant $CaCl_2.2H_2O$ with a concentration of 2.5 g/l. Therefore, the coagulant $CaCl_2.2H_2O$ with a concentration of 2.5 g/l was selected as the convenient coagulant.

**Table 9. Effects of the coagulants on the filtration process.**

| Coagulant | Optimal concentration (g/l) | Cake formation rate (mm/s) | Moisture content (%) | Specific cake resistance (kg/m $\times 10^{12}$) | Resistance to filter cloth ($m^{-1} \times 10^{10}$) | Filtration time (S) | Pressure at the end of dewatering (bar) |
|---|---|---|---|---|---|---|---|
| $CaCl_2 . 2H_2O$ | 2.5 | 0.068 | 12.54 | 3.84 | 60.08 | 600 | 0.7 |
| KCl | 1.5 | 0.057 | 12.31 | 4.60 | 81.91 | 500 | 0.7 |
| $BaCl_2$ | 1.5 | 0.065 | 12.38 | 4.11 | 67.83 | 550 | 0.7 |
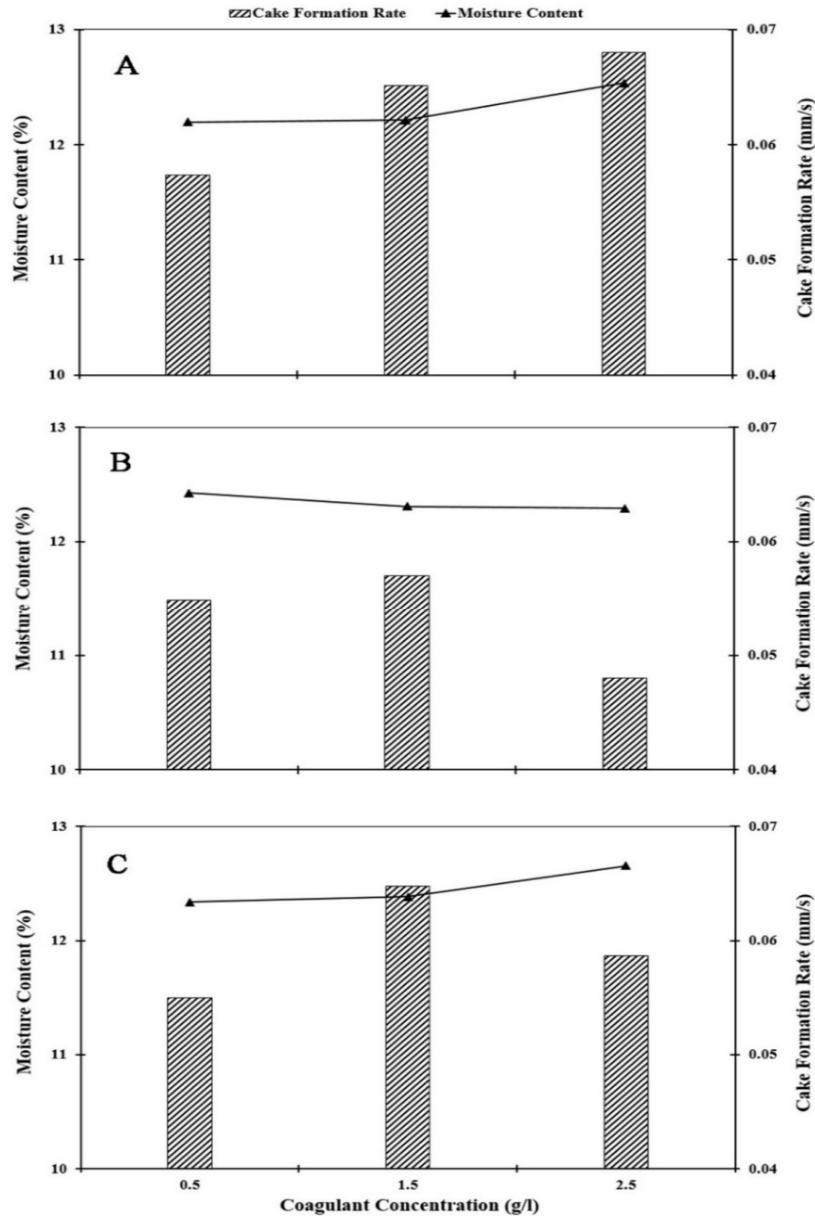


**Figure 14. Effects of coagulants on the moisture content and cake formation rate, $CaCl_2 . 2H_2O$ (A), $KCl$ (B), and $BaCl_2$ (C).**

The results of the filtration tests with and without additives in the optimal conditions are summarized in Table 10. According to this table, the coagulant $CaCl_2 . 2H_2O$ has no meaningful effect on the cake formation rate, resistance to filter cloth, and specific cake resistance. Its result in optimal conditions is similar to the test without any chemical. Therefore, the other three additives

(flocculant A25, surfactant Cop20-101, and perlite as filter aid) in optimal values were considered for the statistical analysis with the Central Composite Design (CCD) methodology.

**Table 10. Results of the filtration tests with and without additives in optimal conditions.**

| Chemicals | Concentration | Cake formation rate (mm/s) | Moisture content (%) | Specific cake resistance (kg/m × 10¹²) | Resistance to filter cloth ($m^{-1} \times 10^{10}$) | Filtration time (s) |
|---|---|---|---|---|---|---|
| Flocculant (A25) | 15 (g/t) | 0.184 | 14.88 | 1.46 | 26.94 | 260 |
| Coagulant ($CaCl_2 . 2H_2O$) | 2.5 (g/t) | 0.068 | 12.54 | 3.84 | 60.08 | 450 |
| Surfactant (Cop20-101) | 3 (cc/l) | 0.12 | 11.22 | 2.65 | 113.21 | 600 |
| Filter aid (Perlite) | 2.5 (%) | 0.17 | 12.65 | 1.82 | 84.41 | 600 |
| Without additive | --- | 0.056 | 13.67 | 4.96 | 61.71 | 550 |

### 3.2. Statistical analysis

The results of the statistical analysis performed by the CCD method and its responses (cake formation rate and cake moisture content) are presented in Table 11.

**Table 11. Results of the statistical analysis performed by the CCD method, coded and actual levels of variables with their responses.**

| Run No. | Coded level of variables | | | Actual level of variables | | | Responses | |
|---|---|---|---|---|---|---|---|---|
| | $X_1$ | $X_2$ | $X_3$ | $X_1$ (g/t) | $X_2$ (%) | $X_3$ (cc/l) | Cake formation rate (mm/s) | Cake moisture content (%) |
| 1 | 0 | +1 | 0 | 13 | 4.69 | 3 | 0.485 | 13.8 |
| 2 | -1 | 0 | 0 | 2.91 | 2.5 | 3 | 0.108 | 12.2 |
| 3 | +α | 0 | 0 | 13 | 2.5 | 3 | 0.210 | 12.2 |
| 4 | +α | 0 | 0 | 13 | 2.5 | 3 | 0.212 | 12.3 |
| 5 | +α | 0 | 0 | 13 | 2.5 | 3 | 0.215 | 12.2 |
| 6 | -α | -α | +α | 7 | 1.2 | 4.5 | 0.116 | 11.3 |
| 7 | 0 | 0 | +1 | 13 | 2.5 | 5.52 | 0.190 | 12.8 |
| 8 | 0 | -1 | 0 | 13 | 0.31 | 3 | 0.170 | 11.3 |
| 9 | 0 | 0 | 0 | 13 | 2.5 | 3 | 0.209 | 12.2 |
| 10 | -α | -α | -α | 7 | 1.2 | 1.5 | 0.145 | 12.1 |
| 11 | +α | +α | +α | 19 | 3.8 | 4.5 | 0.303 | 14.4 |
| 12 | +1 | 0 | 0 | 23.09 | 2.5 | 3 | 0.369 | 14.5 |
| 13 | 0 | 0 | 0 | 13 | 2.5 | 3 | 0.211 | 12.3 |
| 14 | +α | +α | -α | 19 | 3.8 | 1.5 | 0.597 | 16.4 |
| 15 | 0 | 0 | 0 | 13 | 2.5 | 3 | 0.208 | 12.1 |
| 16 | +α | -α | -α | 19 | 1.2 | 1.5 | 0.294 | 15.1 |
| 17 | 0 | 0 | -1 | 13 | 2.5 | 0.48 | 0.251 | 15.8 |
| 18 | -α | +α | -α | 7 | 3.8 | 1.5 | 0.288 | 12.2 |
| 19 | +α | -α | +α | 19 | 1.2 | 4.5 | 0.171 | 11.8 |
| 20 | -α | +α | +α | 7 | 3.8 | 4.5 | 0.185 | 12.3 |

After processing the experimental data by CCD using the "Design Expert 7" software, a model was fitted to each response. The model equations in terms of the coded variables are presented as follow:

$$\text{Ln (cake formation rate)} = -1.57 + 0.32 \times X_1 + 0.31 \times X_2 - 0.083 \times X_3 - 0.069 \times X_1 \times X_3 - 0.044 \times X_2 \times X_3 + 0.11 \times X_2^2 - 0.15 X_1^2 X_3 \quad (6)$$

$$\text{Ln (cake moisture)} = +2.51 + 0.051 \times X_1 + 0.052 \times X_2 - 0.058 \times X_3 - 0.04 \times X_1 \times X_3 + 0.024 \times X_2 \times X_3 + 0.025 X_1^2 + 0.048 \times X_3^2 + 0.038 \times X_1 \times X_2^2 \quad (7)$$

The graph of the predicted values *vs.* the actual values for the cake formation rate and cake moisture content confirmed that the actual values were in good agreement with the predicted values for both responses with an acceptable correlation coefficient ($R^2$ more than 90 % for both responses).

### 3.2.1. ANOVA analysis

The results of the analysis of variance (ANOVA) including the sum of squares, degrees of freedom, mean square, F value, and P value for the cake formation rate and cake moisture content as the main responses of filtration are presented in Tables 12 and 13, respectively. As shown in these tables, the p-value for the model is less than 0.05, which indicates that the model is valid. According to Table 12, the p-value for the parameters $X_1$, $X_2$, $X_3$, $X_1 X_3$, $X_2 X_3$, $X_2^2$, and $X_1^2 X_3$ is also less than 0.05, and therefore, affects the rate of cake formation. These parameters for response of the cake moisture content are $X_1$, $X_2$, $X_3$, $X_1 X_3$, $X_2 X_3$, $X_1^2$, $X_3^2$, and $X_1 X_2^2$ (Table 13).

**Table 12. ANOVA for response of the cake formation rate.**

| Source | Sum of squares | Df | Mean square | *F*-value | *P*-value Prob > *F* | Note |
|--------|---------------|----|-------------|-----------|----------------------|------|
| Model | 3.39 | 7 | 0.48 | 137.79 | <0.0001 | Significant |
| $X_1$ | 1.41 | 1 | 1.41 | 400.34 | <0.0001 | |
| $X_2$ | 1.29 | 1 | 1.29 | 366.9 | <0.0001 | |
| $X_3$ | 0.039 | 1 | 0.039 | 11.03 | 0.0089 | |
| $X_1 X_3$ | 0.038 | 1 | 0.038 | 10.93 | 0.0091 | |
| $X_2 X_3$ | 0.016 | 1 | 0.016 | 4.5 | 0.0629 | |
| $X_2^2$ | 0.16 | 1 | 0.16 | 44.37 | <0.0001 | |
| $X_1^2 X_3$ | 0.078 | 1 | 0.078 | 22.06 | 0.0011 | |
| Residual | 0.032 | 9 | 0.078 | --- | --- | |
| Lack of fit | 0.031 | 7 | $3.515 \times 10^{-3}$ | 15.29 | 0.0627 | Not significant |
| Pure error | $5.803 \times 10^{-5}$ | 2 | $4.436 \times 10^{-3}$ | --- | --- | |
| Cor total | 3.42 | 16 | $2.901 \times 10^{-4}$ | --- | --- | |

**Table 13. ANOVA for response of the cake moisture content.**

| Source | Sum of squares | Df | Mean square | *F*-value | *P*-value Prob > *F* | Note |
|--------|---------------|----|-------------|-----------|----------------------|------|
| Model | 0.21 | 8 | 0.026 | 29.11 | <0.0001 | Significant |
| $X_1$ | 0.015 | 1 | 0.015 | 16.49 | 0.0036 | |
| $X_2$ | 0.037 | 1 | 0.037 | 40.91 | <0.0002 | |
| $X_3$ | 0.046 | 1 | 0.046 | 50.66 | 0.0001 | |
| $X_1 X_3$ | 0.013 | 1 | 0.013 | 13.84 | 0.0059 | |
| $X_2 X_3$ | $4.661 \times 10^{-3}$ | 1 | $4.661 \times 10^{-3}$ | 5.15 | 0.0529 | |
| $X_1^2$ | $7.478 \times 10^{-3}$ | 1 | $7.478 \times 10^{-3}$ | 8.27 | 0.0207 | |
| $X_3^2$ | 0.029 | 1 | 0.029 | 31.82 | 0.0005 | |
| $X_1 X_2^2$ | $4.895 \times 10^{-3}$ | 1 | $4.895 \times 10^{-3}$ | 5.41 | 0.0484 | |
| Residual | $7.235 \times 10^{-3}$ | 8 | $9.044 \times 10^{-4}$ | --- | --- | |
| Lack of fit | $7.101 \times 10^{-3}$ | 6 | $1.183 \times 10^{-3}$ | 17.61 | 0.0547 | Not significant |
| Pure error | $1.344 \times 10^{-4}$ | 2 | $6.719 \times 10^{-5}$ | --- | ---- | |
| Cor total | 0.22 | 16 | --- | --- | ---- | |

### 3.2.2. Influence of variables

The effects of the variables on both responses (cake formation rate and cake moisture content) were also investigated. According to Figures 15A, 15B, and 15C, which show the effects of the variables on the cake formation rate, increasing the amount of filter aid and the concentration of the flocculant increased the cake formation rate. Also by increasing the concentration of the surfactant, the cake formation rate reduced. The reason for this phenomenon can be attributed to an excessive increase in the viscosity of the pulp at higher surfactant concentrations. Figures 15D, 15E, and 15F show the effects of the variables on the response of the cake moisture content. Based on the results obtained, by increasing the amount of the filter aid and concentration of the flocculant, the moisture content of the filtered cake increased, which could be attributed to the increase in the porosity in a higher viscosity environment (due to the presence of flocculant and surfactant) and trapped water between the cavities. By increasing the concentration of the surfactant, the filter cake moisture shows a downward trend (Figures 15D, 15E, and 15F). It should be noted that the reduction in moisture to its values in the concentration of 3.75 cc/l shows an exponential trend. From the concentration of 3.75 cc/l to 4.5 cc/l of the tested surfactant, the moisture content of the filter cake was constant and did not decrease.
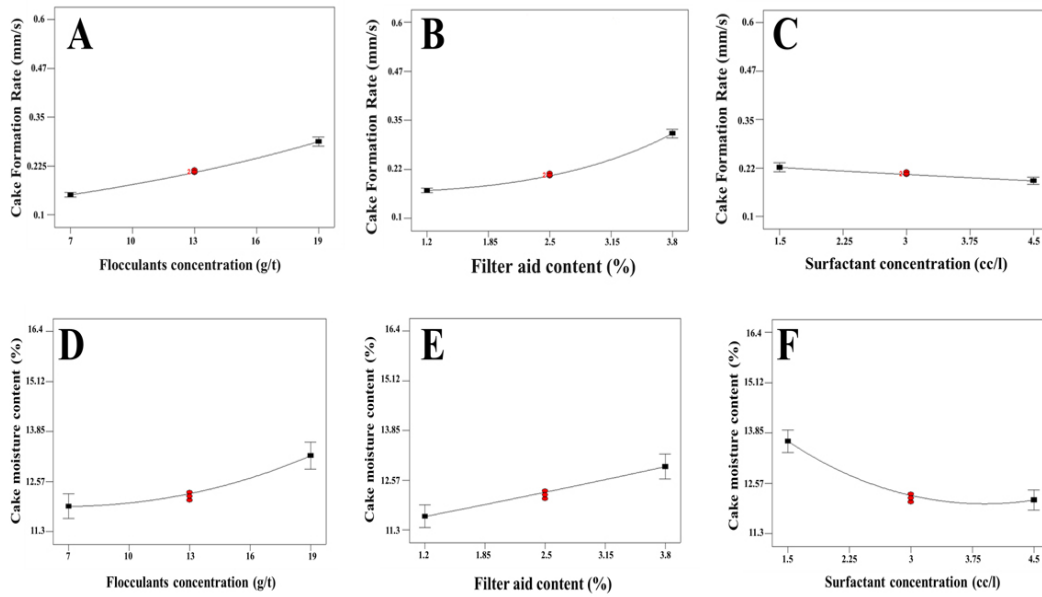
**Figure 15. Effects of the variables on the cake formation rate (A, B, and C) and cake moisture content (D, E, and F) as two main responses.**

Figure 16A shows the effect of the interaction between the concentrations of the flocculant and the surfactant on the cake formation rate when the amount of filter aid is at its mid-level. At a surfactant concentration of 1.5 cc/l, by increasing the concentration of the flocculant, the cake formation rate also increases. The trend of increasing the cake formation rate at concentrations of the tested flocculant in more than 13 g/t also increased. The reason for this phenomenon can be attributed to the forming the bigger clots. When the concentration of surfactant was 4.5 cc/l, the cake formation rate also increased with increase in the flocculant concentration. However, due to the higher viscosity of the pulp (because of the effect of the surfactant viscosity and the filter aid in high concentrations), this trend was not the same as the previous one, and at concentrations above 16 g/t, the process of increasing the cake formation rate stopped. Considering the concentration of the flocculant at the mid-level and concentration of 1.5 cc/l for the surfactant, the increase in the perlite content had a positive effect on the cake formation rate. Similarly, this trend was true when the surfactant was considered at a concentration of 4.5 cc/l but the initial and final points of the cake formation rate graph were lower than those for the previous state (Figure 16B). The reason for this behavior is the reduction in porosity in a higher viscosity of the pulp. Due to the high viscosity,

water is trapped in the cavities due to the addition of a filter aid, which reduces the cake formation rate and increases the moisture content of the filter cake.

The effect of the interaction of the surfactant and flocculant concentrations on the cake moisture content is also presented in Figure 16C. When the amount of filter aid is at the mid-point and the concentration of surfactant is at its lowest level, by increasing the flocculant concentration, the moisture content of the filter cake increases significantly. This phenomenon occurs due to an increase in the size of the clots formed in high concentrations of the flocculant and trapping of water between these clots. On the other hand, when the concentration of the surfactant is high (4.5 cc/l), increasing the concentration of the flocculant has no significant effect on the increase in the moisture content, which is due to a decrease in the surface tension and discharge of water from pulp clots. Figure 16D shows the interaction effect of the amount of filter aid and the concentration of surfactant. When the concentration of the flocculant is at its mid-level, increasing the amount of filter aid in the maximum and minimum surfactant concentrations increases the moisture content of the filter cake. According to the data presented in Figures 16C and 16D, the best results are achieved when the amounts of the filter aid and surfactant concentrations are at low values.
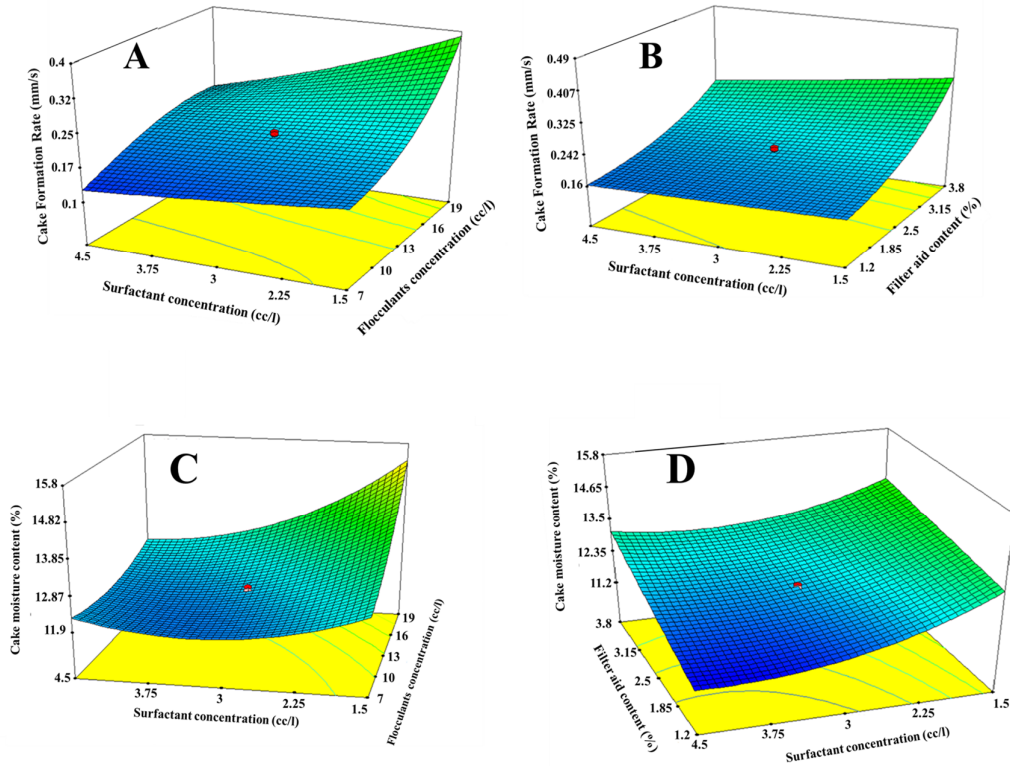
**Figure 16. Effect of interaction of the flocculant and surfactant concentrations (A) and filter aid and surfactant concentrations (B) on the response of the cake formation rate, and effect of interaction of the flocculant and surfactant concentrations (C), and filter aid and surfactant concentrations (D) on the response of the cake moisture content.**

### 3.3.3. Optimization of process variables

Optimization of the results obtained was performed using the DX7 software with the aim of maximizing the response of the cake formation rate and minimizing the response of the filter cake moisture content. The optimization results are presented in Table 14. In order to validate the model, two tests were repeated in the optimal conditions. The results of the validation experiments are presented in Table 15. According to these results, the proposed model well-predicts the filtration process.

**Table 14. Optimal conditions proposed using the DX7 software.**

| Row | Flocculant concentration (g/t) | Filter aid content (%) | Surfactant concentration (cc/l) | Predicted cake formation rate (mm/s) | Predicted cake moisture (%) | Desirability |
|---|---|---|---|---|---|---|
| 1 | 11.68 | 3.8 | 2.92 | 0.297 | 12.7 | 0.64 |
| 2 | 11.73 | 3.8 | 2.94 | 0.297 | 12.71 | 0.64 |
| 3 | 11.61 | 3.8 | 2.92 | 0.269 | 12.69 | 0.64 |
| 4 | 11.82 | 3.8 | 2.97 | 0.298 | 12.72 | 0.64 |

**Table 15. Results of the validation tests in the optimal conditions.**

| Row | Flocculant concentration (g/t) | Filter aid content (%) | Surfactant concentration (cc/l) | Cake formation rate (mm/s) | Cake moisture content |
|---|---|---|---|---|---|
| 1 | 11.68 | 3.8 | 2.92 | 0.273 | 12.3 |
| 2 | 11.73 | 3.8 | 2.94 | 0.269 | 12.5 |

### 4. Conclusions

The aim of this work was to investigate the effects of the additives such as the flocculant, coagulant, surfactant, and filter aid on the filtration responses including the cake formation rate, cake moisture content, specific cake resistance, resistance of filter cloth, and water content recovery. The vacuum top-feed method was applied for dewatering the copper

concentrate. In this work, for the first time, the effect of interaction of the effective additives as the chemical reagents on the filtration process was investigated. The results of the experiments showed that the anionic flocculant A25 with a concentration of 15 g/t, perlite solid filter aid with a content of 2.5%, surfactant Cop20-101 with a concentration of 3 cc/l, and coagulant $CaCl_2.2H_2O$ with a concentration of 2.5 g/l had the best results. On the other hand, due to the positive surface charge of the particles in the pH of the environment, the anionic flocculants were found to have a better effect on the filtration process than the cationic flocculants. The effective additives including the flocculant, surfactant, and filter aid were analyzed statistically using the DX7 software and by the Central Composite Design (CCD) method with the aim of maximizing the cake formation rate and minimizing the moisture content of filter cake. According to the ANOVA analysis, all the three additives flocculant, surfactant, and filter aid are effective on the cake formation rate and cake moisture content. Under the optimal conditions and taking 11.68 g/t of the flocculant A25, 3.8% perlite as solid filter aid, and 2.92 cc/l of the surfactant Cop2-101, responses of the cake formation rate and cake moisture content were reported to be 0.297 mm/s and 12.7 %, respectively. By performing the validation tests, it was found that the model presented using the DX7 software well-described the filtration process.

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## References

[1]. Ihle, C.F. and Kracht, W. (2018). The relevance of water recirculation in large scale mineral processing plants with a remote water supply. Journal of Cleaner Production. 177: 34-51.

[2]. Gálvez, E.D., Cruz, R., Robles, P.A. and Cisternas, L.A. (2014). Optimization of dewatering systems for mineral processing. Minerals Engineering. 63: 110-117.

[3]. Usher, S.P. and Scales, P.J. (2005). Steady state thickener modelling from the compressive yield stress and hindered settling function. Chemical Engineering Journal. 111(2-3): 253-261.

[4]. Northey, S.A., Mudd, G.M., Werner, T.T., Jowitt, S.M., Haque, N., Yellishetty, M. and Weng, Z. (2017). The exposure of global base metal resources to water criticality, scarcity and climate change. Global Environmental Change. 44: 109-124.

[5]. Aljerf, L. (2018). Data of thematic analysis of farmer's use behavior of recycled industrial wastewater. Data in Brief. 21: 240-250.

[6]. Bürger, R., Concha, F. and Karlsen, K.H. (2001). Phenomenological model of filtration processes: 1. Cake formation and expression. Chemical Engineering Science. 56(15): 4537-4553.

[7]. Qi, Y., Thapa, K.B. and Hoadley, A.F. (2011). Application of filtration aids for improving sludge dewatering properties-a review. Chemical Engineering Journal. 171(2): 373-384.

[8]. Wills, B.A., and Finch, J. (2015). Wills' mineral processing technology: an introduction to the practical aspects of ore treatment and mineral recovery. Butterworth-Heinemann.

[9]. Adrian, H. and Meintjes, C. (2001). Dry tailings disposal-increased production and potential water saving. Mine Waste Journal.

[10]. Wang, Q., Lin, X. and Chen, D.R. (2016). Effect of dust loading rate on the loading characteristics of high efficiency filter media. Powder Technology. 287: 20-28.

[11]. Luo, H., Ning, X.A., Liang, X., Feng, Y. and Liu, J. (2013). Effects of sawdust-CPAM on textile dyeing sludge dewaterability and filter cake properties. Bioresource technology. 139: 330-336.

[12]. Christensen, M.L., Johansson, C., Sedin, M. and Keiding, K. (2011). Nonlinear filtration behavior of soft particles: Effect of dynamic cake compression. Powder technology. 207 (1-3): 428-436.

[13]. Guo, D., Wang, H., Fu, P., Huang, Y., Liu, Y., Lv, W. and Wang, F. (2018). Diatomite precoat filtration for wastewater treatment: Filtration performance and pollution mechanisms. Chemical Engineering Research and Design. 137: 403-411.

[14]. Ripperger, S., Gosele, W., Alt, C. and Loewe, T. (2013). Filtration, 1. Fundamentals, in: Ullmann's Encyclopedia of Industrial Chemistry. On-line ed., Wiley.

[15]. Sparks, T. (2012). Solid-liquid filtration: Understanding filter presses and belt filters. Filtration & Separation. 49 (4): 20-24.

[16]. Tarleton, S. and Wakeman, R. (2006). Solid/liquid separation: equipment selection and process design. Elsevier.

[17]. Qi, Y., Thapa, K.B. and Hoadley, A.F. (2011). Application of filtration aids for improving sludge dewatering properties-a review. Chemical Engineering Journal. 171(2): 373-384.

[18]. Dong, K.J., Zou, R.P., Yang, R.Y., Yu, A.B. and Roach, G. (2009). DEM simulation of cake formation in sedimentation and filtration. Minerals Engineering. 22 (11): 921-930.

[19]. Ni, L.A., Yu, A.B., Lu, G.Q. and Howes, T. (2006). Simulation of the cake formation and growth in cake filtration. Minerals engineering. 19 (10): 1084-1097.

[20]. Day, A. (2002). Mining Chemicals Handbook, Revised Edition. CYTEC, Horton Printing Company, Meriden, CT.

[21]. Patra, A.S., Makhija, D., Mukherjee, A.K., Tiwari, R., Sahoo, C.R. and Mohanty, B.D. (2016). Improved dewatering of iron ore fines by the use of surfactants. Powder Technology. 287: 43-50.

[22]. Castro, S. and Laskowski, J.S. (2015). Depressing effect of flocculants on molybdenite flotation. Minerals Engineering. 74: 13-19.

[23]. Fan, Y., Dong, X. and Li, H. (2015). Dewatering effect of fine coal slurry and filter cake structure based on particle characteristics. Vacuum. 114: 54-57.

[24]. Garmsiri, M.R. and Shirazi, H.H.A. (2014). The effect of grain size on flocculant preparation. Minerals Engineering. 65: 51-53.

[25]. Sarkar, A.K., Ghorai, S., Patra, A.S., Mishra, B.K., Mandre, N.R. and Pal, S. (2015). Modified amylopectin based flocculant for the treatment of synthetic effluent and industrial wastewaters. International journal of biological macromolecules. 72: 356-363.

[26]. Liu, H., Yang, X., Zhang, Y., Zhu, H. and Yao, J. (2014). Flocculation characteristics of polyacrylamide grafted cellulose from Phyllostachys heterocycla: an efficient and eco-friendly flocculant. Water research. 59: 165-171.

[27]. Wang, L.F., He, D.Q., Tong, Z.H., Li, W.W. and Yu, H.Q. (2014). Characterization of dewatering process of activated sludge assisted by cationic surfactants. Biochemical engineering journal. 91: 174-178.

[28]. Lihong, D., Xu, C., Wenping, L. and Qixin, Z. (2011). A study on enhancement of filtration process with filter aids diatomaceous earth and wood pulp cellulose. Chinese Journal of Chemical Engineering. 19(5): 792-798.

[29]. Wang, J.P., Chen, Y.Z., Yuan, S.J., Sheng, G.P. and Yu, H.Q. (2009). Synthesis and characterization of a novel cationic chitosan-based flocculant with a high water-solubility for pulp mill wastewater treatment. Water research. 43(20): 5267-5275.

[30]. Dias, C.L.P., Valadão, G.E.S., Araujo, A.C., Peres, A.E.C. and Amarante, S.C. (2003). The effect of reagents on ultrafine iron ore vacuum filtration. Filtration & separation. 40(5): 36-39.

[31]. Fiol, N. and Villaescusa, I. (2009). Determination of sorbent point zero charge: usefulness in sorption studies. Environmental Chemistry Letters. 7(1): 79-84.

[32]. Yang, Z., Yuan, B., Huang, X., Zhou, J., Cai, J., Yang, H., Li, A. and Cheng R. (2012). Evaluation of the flocculation performance of carboxymethyl chitosan-graft-polyacrylamide, a novel amphoteric chemically bonded composite flocculant. Water Research. 46(1): 107-114.

[33]. Simate, G.S., Iyuke, S.E., Ndlovu, S. and Heydenrych, M. (2012). The heterogeneous coagulation and flocculation of brewery wastewater using carbon nanotubes. Water Research. 46(4): 1185-1197.

[34]. Singh, B.P. (1997). The influence of surface phenomena on the dewatering of fine clean coal. Filtration & separation. 34(2): 159-163.

[35]. Moudgil, B.M. (1986). Selection of flocculants for solid-liquid separation processes. Advances in Solid-Liquid Separation.

[36]. Besra, L., Singh, B.P., Reddy, P.S.R. and Sengupta, D.K. (1998). Influence of surfactants on filter cake parameters during vacuum filtration of flocculated iron ore sludge. Powder technology. 96(3): 240-247.

[37]. Mackenzie, M., Malhotra, D. and Riggs, W.F. (1986). Chemical Reagents in the mineral Processing Industry. Society of Mineral Engineering. Colorado, USA. 139-145.

[38]. Fatahi, M., Noaparast, M. and Shafaei, S.Z. (2014). Nickel extraction from low grade laterite by agitation leaching at atmospheric pressure. International Journal of Mining Science and Technology. 24(4): 543-548.

# بررسی تاثیر فلوکولانت، کواگولانت، سطح‌ساز و کمک فیلتر بر کارآیی فرآیند فیلتراسیون کنسانتره مس: مکانیزم و بهینه‌سازی

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**چکیده:**

در چند سال اخیر بحران کم‌آبی به دلایل متعددی به ویژه استفاده نادرست و بی‌رویه از منابع آب، به چالشی بزرگ تبدیل شده است. اکثر روش‌های کانه‌آرایی در محیطی که محتوی مقدار قابل توجهی آب است، انجام می‌شوند. در این صورت محصول پر عیار شده نهایی، به صورت پالپی بوده که گاهی دارای رقت خیلی زیاد است و باید با آبگیری، رطوبت آن را به حدی کاهش داد که برای حمل یا عملیات صنعتی دیگر مناسب باشد. فیلتراسیون به عنوان یکی از فرآیندهای مهم در بازیابی آب، یک عملیات واحد است که طی آن، ذرات معلق جامد از مایع جدا می‌شوند. در تحقیق حاضر، تأثیر مواد افزودنی مؤثر بر فرآیند فیلتراسیون بر پاسخ‌هایی از جمله مقاومت پارچه فیلتر (R)، مقاومت مخصوص کیک ($\alpha$)، میزان رطوبت، نرخ بازیابی آب و نرخ تشکیل کیک به روش خوراک‌دهی از بالا مورد بررسی قرار گرفتند. در انجام آزمایش‌ها، پارامترهایی نظیر فلوکولانت، کمک فیلتر، کواگولانت و سطح‌ساز به صورت یک عامل در زمان و تحلیل آماری بررسی شدند. نتایج بهینه حاصله از انجام آزمایش‌ها بصورت یک عامل در زمان، عبارتند از: فلوکولانت A25 با غلظت 15 (g/t)، کمک فیلتر پرلیت با مقدار 2/5 (٪)، سطح‌ساز -Cop20 101 با غلظت 3 (cc/l) و کواگولانت $CaCl_2 2H_2O$ با غلظت 2/5 (g/l). پارامترهای فلوکولانت، سطح‌ساز و کمک فیلتر به‌صورت تحلیل آماری با هدف بیشینه کردن نرخ تشکیل کیک و کمینه کردن رطوبت کیک فیلتر مورد بررسی قرار گرفتند. تحت شرایط بهینه و با در نظر گرفتن 11/68 (g/t) فلوکولانت A25 ، 3/8 (٪) کمک فیلتر پرلیت و 2/92 (cc/l) سطح‌ساز Cop20-101، نرخ تشکیل کیک 0/297 (mm/s) و کیک فیلتر با رطوبت 12/7 (٪) حاصل شد.

**کلمات کلیدی:** فیلتر خلاء، رطوبت کیک، بازیابی آب، نرخ تشکیل کیک، رطوبت کیک فیلتر.