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Treatment of Turbid Wastewater by Means of Clean Electrochemical Technology: Investigation of the New Operational Parameters

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Abstract

Two different turbid wastewater (oily and milky) effluents were treated applying electrocoagulation process with aluminum / aluminum electrodes. The effect of operational parameters such as initial pH, current density, reaction time, initial concentrations (1000, 10000 ppm equivalent to 0.1-1 wt%), ionic strength, electrical energy consumption, types of circuits(direct current(DC), pulse current (PC) and alternating pulse current(APC)), were investigated to reach highest turbidity removal efficiency. The optimum parameters to reach highest (99.9 %) turbidity removal efficiency were determined as follows: EEC range: 2.3-14.7 kwh/m³ wastewater, CD: 90-194.5 A/m², operating time: 7-22 min, pH: 3-9 (optimum pH = 7 for oily waste waters and optimum pH = 5 for milky waste waters), Voltage: 20 volts. Similar removal efficiency was obtained in either DC or PC. However in "PC", water recovery (0.93m³/m³ wastewater)was significant at minimum energy consumption (turbidity is less than 5 NTU). COD (chemical oxygen demand) reduction was more than 90%.

Keywords: *Turbidity, Electrocoagulation (EC); Milky and Oily Wastewater, Pulse Current (square wave), Alternating Pulse Current.*

Introduction

Organicparticles, oil, algae, colloidal and soluble substances present in water often initiate turbidity or color in waste waters. Small particles with density close to water may never settle and stabilize in the water. However coagulation and flocculation of these particles into larger flocs is the necessary step for their removal followed by sedimentation [1]. In this regard, electrochemical treatment of turbid water was proceeding as an effective method to increase the clarity and purity of water [2]. Sacrificial anodes made of iron or aluminum are known to be used [3]. Dissolved anode introduces coagulating ions of $A1^{3+}$ in the turbid water suspension. These ions coagulate with turbidity agents existing in water in a similar manner to the addition of coagulation

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chemicals such as alum and ferric chloride and allow easier removal of the pollutants [4]. Usually aluminum is the preferred material since it does not induce color to the supernatant, and the electrocoagulation unit is environmentally friendly [5]. Oily wastewater has caused serious problem in various industries for decades. Industrial sources of oily wastewater include petroleum refining, metals manufacturing and machining, as well as food processing. In this respect, American Petroleum Institute (API) has exerted a great deal of effort to develop processes to treat such oily waste waters. Based on the design parameters published by the American Petroleum Institute, the standard apparatus in refinery wastewater treatment is the API Separator, but treated effluents from this unit may still contain 400-500 mg/L of oil. The wastewater from metal working processes contains grinding and cutting oil, lubricant fluids and cooling oil water emulsions with the oil load in the range between 100-5000 mg/L. Electrochemical processes to treat oily wastewater were first described in patent literature in 1903 [6-11].

Description of EC technique

The most widely used electrode materials in EC process are aluminum (Al) and iron. In the case of aluminum, the mechanism and main reactions are described by Mollah and Morkovsky [12].The electrolytic dissolution of Al anode at low pH produces Al^{3+} and $Al(OH)_2^+$, which is then transformed to gelatinous ball $Al(OH)_3$, that finally polymerized to Al_p (OH)_{3p}[2]:

 $Al \rightarrow Al^{3+} + 3e^{-} \tag{1}$

$$Al^{3+}+3H_2O \rightarrow Al (OH)_3+3H^+$$
(2)

$$nAl(OH)_3 \rightarrow Al_n(OH)_{3n}$$
 (3)

Gelatinous and charged species effectively adsorbs organic matters on the floc surface or precipitates during EC process. Additionally, both anode and cathode will dissolve chemically.Note that, the dissolution of aluminum is thermodynamically favored and it should proceed spontaneously. Gelatinous and charged species effectively remove oil by adsorption on the floc surface or by enmeshment precipitating during EC process.

In this study, a wide range of initial concentrations (1000, 10000 ppm) were investigated, whereas other studies had focused on initial concentrations up to 5000 ppm [4,6,8]. Also in this investigation, PC and APC modes for removal of organic matter were utilized, in addition to traditional DC method. Therefore, the operational parameters corresponding to PC and APC modes were optimized. Two processes that can interfere with the electrolytic process are polarization or passivity and mass transport control [12-14]. It is to be noted that in this investigation the influences of PC and

APC and various supporting electrolyte were studied to avoid electrode passivity. In addition "water recovery" is introduced and calculated in order to show the feasibility of recycling the treated water.

Experimental

Material and Methods

Test fluids consisting of oil-in-water emulsion and milk-powder in water were selected. Oilconcentrated cutting fluid containing 70% mineral oil, 12% surfactant 8% cosurfactant and 10% additive was used as oil emulsion, and was purchased as commercial cutting oil, Z1 (manufactured by Behran oil company-IRAN). All chemicals were of analytical grade and purchased from Merck-Germany. The oilin- water emulsions were prepared by adding the concentrated oil to deionized water at room temperature applying magnetic stirring at 500 rpm for 10 min. Solutions of varying concentrations (1000-10000 ppm (mg/L) equivalent to 0.1-1 wt% (similat to the industrial applications of oil in emulsion) were prepared. Their initial turbidity was measured as 1024-12100 NTU(nephelometer turbidity unit).Other synthetic wastewater samples were made from milk powder. Initial concentrations of milky waste waters were 1000-10000 ppm equivalent to 0.1-1 wt%, and their initial turbidity was 620-6300 NTU(Table 1).

Table 1. Propertie	s of initial waste waters

Kind of wastewater	Initial concentration $C_0(ppm)$ Initial pH		NaCl concentration (kg/m ³) as supporting electrolyte	Initial specific conductivity (NaCl) γ :(mS/cm)	Initial turbidity (NTU)
Oily	1000*	7	0.57	1.26	1024
	10000	7	0.86	1.88	12100
Milky	1000**	5	0.46	1.28	620
	10000	5	0.71	1.58	6300

* Initial COD for 1000 ppm=6500mg/L

** Initial COD for 1000 ppm=900mg/L

The conductivity of each sample was adjusted to the desired level by adding an appropriate amount of electrolytes (NaCl, NaNO₃, KCl, Poly (Aluminum Chloride)). The initial parameters of the emulsions are presented in Table 1.

The electrocoagulation experiments are carried out in one liter batch using cylindrical glass cell (Figure 1).



Figure 1. Electro coagulation reactor [15].

All experiments were performed at room temperature (20– 22° C), Two tasks have

been considered to control the passivity and turbidity removal efficiency, are:

1) the influence of ionic strength effect on some supporting electrolytes such as NaCl, KCl, PAC and NaNO₃, was examined and compared with each other to achieve higher removal efficiency, 2) Two types of circuit, PC and APC, have been exerted to reduce the polarization and the sludge resistance around the electrodes and hitches the sludge from anode and cathode. Initial solution conductivity ways increased adding proper amounts of NaCl, KCl, PAC and NaNO₃ before each experiment (Table 1). The effect of initial pH on turbidity removal efficiency is evaluated using sulfuric acid solution and sodium hydroxide (0.1 M). Experiments were performed in a batch system (Figure 1.). Net volume of reactor was 0.7 L and the effective area of each electrode [Al (cathode) and Al (anode)] used was 36cm^2 (6 cm × 6 cm) and the gap between electrodes was 1.5 cm. One DC power supply package having an input of 220Volt and variable output of 0-30 volt, with variable current 0-4 ampere was used. An Electro alternative pulse current (square wave) generator was applied to study the impact of circuit type (from Nano PusheshFelez, Iran). The optimum operating conditions are described below.

At the end of EC experiment, the incurred precipitate was removed by filtration and the turbidity of treated wastewater (or output) was determined before and after filtration in each batch and compared with each other (For turbidity removal efficiency calculations) and analyzed. Once the optimal conditions were reached the raw wastewater and treated wastewater samples were filtered and analyzed for COD and turbidity. All experiments were repeated twice, and the experimental error was about 4%.

Chemical Analysis

The emulsion turbidity was measured (based on method 2130-B standard methods [16], using a Hach-Lange turbidity meter with a highest sensitivity of 0.01 NTU. The removal efficiency (%Turbidity removal) was calculated using the following equation:

Turbidity removal efficiency (%) =
$$\frac{T_0 - T}{T_0} \times 100$$
(4)

T_0 : Initial turbidity (before EC), T: Final turbidity (after EC) in NTU.

In all experiments, the pH was measured by metrohm pH meter model 827, the conductivity was measured (based on method 2510-B [16]by Hach-Lange conductivemeter, chemical oxygen demand (COD) was determined by conventional potassium dichromate [16], the spectrophotometer was Hach-Lange(DR/2800). Power Consumption or Electrical Energy Consumption

(EEC), (kwh/m³ wastewater)= $V \times I \times t/v$ (5)

V: applied voltage (volt), I: working amperage(ampere), t (or t_{EC})=time of Electrocoagulation (min), v: volume of wastewater (m³).

The current density (CD) was calculated from the equation 6 as follows:

$$CD = \frac{I(A)}{2S_{electrode}(m^2)}$$
(6)

Where I: current (A), S:surface area of the

electrode (m²).

Water recovery is the new operational parameter which is introduced in this study as follows:

Water recovery=
$$\frac{volume \ of \ generated \ water(after EC)}{initial \ volume \ of \ wastewater(befor EC)}$$
(7)

Indeed the supernatant that remained above the dense sludge after EC was named "generated water" or treated water, lost its organic matter and sludge during EC process. Water recovery is calculated and the data are summarized in Tables 2 and 3 and compared in APC, DC and PC states.

 Table 2. Optimized operational parameters of different waste waters (current type:DC, supporting electrolyte: NaCl, mixing:120 rpm).

Type of circuitInitial solution concentratin C_0 (ppm)Current density $(A/m2)$ (Eq.6)	Current	Operating time(min)		Energy	Turbidity	Turbidity	Water recov-	
	Initial solution concentratin C_0 (ppm)	density (A/m2) (Eq.6)	With mixing	Without mixing	consumption (kwh/m ³ waste water) (Eq.5)	removal removal efficiency (%) efficiency (%) before filtration (Eq.4) (Eq.4)	ery (m ³ /m ³ waste water) (Eq.7)	
Oily	1000	90	10	30	3.09	99.5	99.9	0.93
waste	10000	161	22	37	12.15	99.8	99.9	0.86
Milky	1000	139	8	18	3.81	99.2	99.5	0.93
waste	10000	194.5	22	30	14.66	99.7	99.9	0.78

 Table 3.Optimized operational conditions of oily waste waters under different electrolysis conditions, supporting electrolyte; NaCl.

			5			
Type of circuit	Initial oil solution concentration C0 (ppm)	Current density (A/m ²) (Eq.6)	Operating time (min)	Energy consumption (kwh/m ³ wastewater) (Eq.5)	Turbidity removal efficiency (%) after simple filtration (Eq.4)	Water recovery (m ³ /m ³ waste water) (Eq.7)
Direct current	1000	90	10	3.09	99.9	0.93
	10000	161	22	12.15	99.9	0.86
Pulse current	1000	90	7.5	2.32	99.4	0.93
	10000	161	13.5	7.45	99.9	0.86
Alternating	1000	90	10	3.09	99.9	0.93
pulse current	10000	161	22	12.15	99.9	0.86

Results and Discussion

Effect of current density on the turbidity removal efficiency

In all electrochemical processes, current

density (CD) is the most important parameter to control reactions in the reactor. According to Faraday's law, CD determines the coagulant generation rate and regulates the rate and size of the bubble and the growth of flocks [12]. A series of electrocoagulation tests were carried out by solutions containing constant initial concentrations (either 1000 or 10000 ppm) applying current densities varied from 50 to 220 A/m^2 . It was discovered that, higher CD enhances the turbidity removal efficiency (Figure 2).



Figure 2. Effect of current density on the turbidity removal efficiency ($C_0=1000$ ppm (milky wastewater), $t_{rc}=8$ min, electrolyte:NaCl.

Figure 2 shows the turbidity removal efficiency versus different CD (for example, 1000 ppmmilk). The results show improvement over previous works [17-19]. Other optimized CD has been presented in Tables 2 to 4 for defined conditions achieving higher removal efficiency. It should be noted that, most of the previous studies used initial concentration up to 1000 ppm [17-19]. However in our study, optimized current densities are introduced to remove impurity from higher concentrations (10000ppm) solutions based on maximum removal efficiency (99.9%). The reported results in the literature are based on Al/Fe [19] and metal/steel electrodes [15] whereas Al/Al electrodes were used in this work.

 Table 4. Optimized operational conditions for milky waste waters under different electrolysis conditions, supporting electrolyte: NaCl.

Type of circuit	Initial milky waste waterC ₀ (ppm)	Optimum cur- rent density (A/ m2) (Eq.6)	Operating time(min)	Energy consump- tion (kwh/m ³ waste- water)	Turbidity removal efficiency (%) after simple filtration (Eq.4)	Water recovery (m ³ /m ³ waste water) (Eq.7)
Direct current	1000	139	8	(Eq.5) 3.81	99.5	0.93
	10000	194.5	22	14.66	99.9	0.78
Pulse current	1000	139	7	3.33	99.3	0.93
	10000	194.5	22	14.66	99.8	0.78
Alternating	1000	139	8	3.81	99.3	0.93
pulse current	10000	194.5	22	14.66	99.8	0.78

Effect of electrolysis time on the turbidity removal efficiency

Operating time also influences the turbidity removal efficiency in EC process. Actually reaction time determines the production rate of Al²⁺or Al³⁺ ions from electrodes. As is observed in Figures 3 and 4, turbidity removal efficiency was above 99% during the first 8 minutes and then reached almost constant rate. Therefore, the optimum time for maximum turbidity removal efficiency was 8 minutes. These results are also based on minimum energy consumption and maximum turbidity removal efficiency.



Figure 3. Effect of time on the turbidity removal efficiency ($C_0=1000$ ppm (milky waste waters), CD=139A/m², supporting electrolyte: NaCl).



Figure 4. Effect of time on the turbidity removal efficiency ($C_0=1000$ ppm (oily waste water), CD=90A/m², supporting electrolyte: NaCl).

Effect of initial pH on the turbidity removal efficiency

It has been acknowledged that the pH has important role on the performance of EC process. In electrochemical processes, not only, alkaline and neutral conditions but also high acidic range has been used to remove turbidity [1-15, 17-21]. However it is still unclear that if low or high pH medium is suitable to reach good overall performance in EC process. In this study pH was adjusted, while all of the other parameters were kept constant at optimum condition. Turbidity removal efficiency at various initial pHare shown in Figures 5 and 7.



Figure 5. Effect of initial pH on the turbidity removal efficiency ($C_0=1000$ ppm (oily waste water), $t_{EC}=10$ min,CD=90A/m², supporting electrolyte: NaCl).



Figure 6.Initial pH versus final pH ($C_0=1000$ ppm (oily waste water), $t_{EC}=10$ min,CD=90 A/m², supporting electrolyte: NaCl).



Figure 7.Effect of initial pH on the turbidity removal efficiency (C_0 =1000 ppm (milky waste water), t_{EC} =8 min, CD=139A/m², supporting electrolyte: NaCl).

These results are based on minimum energy consumption and maximum turbidity removal efficiency. Several optimized conditions are presented in Table 2-4.Also see Figures 6 and 8.



Figure 8. Initial pH versus final pH ($C_0=1000$ ppm (mikly waste water), $t_{EC}=8min,CD=139A/m^2$, supporting electrolyte: NaCl).

Type of circuit and its effect on EC- Pulse current (square wave)- Alternating pulse current-Direct current

During EC process while sludge is produced, polarization and fouling phenomena occur around anode. In other words, passivity and mass transport control can interfere with the electrolytic process. For some systems, any increase in current does not lead to a corresponding increase in electrolysis of the electrode. Passivity is caused by buildup of metal oxides on the surface of the electrodes leading to an increase in the resistance to the electrolytic process [12-14].

In this study as a technique, pulse current (PC) (or square wave) and alternating pulse current (APC) have been established and proposed to control the passivity. Figure 10 exhibits applied APC, where in APC state, cathode or anode is typically operating for 2 minutes and then it is replaced or interchanged until EC is completed.However in PC state, cathode and anode are typically operating for 1 minute, then they are shouted down or terminated for 1 min, until EC is completed, $(t_{on}=1 \text{ min}, t_{off}=1 \text{ min}, t_{EC}=\sum t_{on})$



Figure 9. Schematic diagram for pulse current (PC) (or square wave). Cathode and anode are operating for 1 minute, then they are shouted down or quieted for 1 min, until EC is completed,



Figure 10. Schematic diagram for alternating rectangular pulse current (APC) (alternating square wave). Cathode or anode is typically operating for 2 minutes, and then it is replaced or interchanged until EC is completed.

The proposed time was optimized following a series of experiments. Applied voltage was 20 volts in each batch. Equal turbidity removal efficiency is obtained in direct current and alternating pulse current and pulse current. In PC mode, reaction time and therefore energy consumption is at lowest value. However, because of changing electrodes in APC and PC, sludge was hitched around the electrodes and fouling phenomena was limited. Dewatered and dense sludge impressed our experimental team, and water recovery (Eq.7) was significant, at about 0.92m³/m³ wastewater (see Table 2-4 for detailed operational parameters). Turbidity of water recovery in APC and PC and DC mode are under 5 and 10 and 20 NTU respectively after EC process. This is the first time that PC and APC are used for removal of turbidity. Mao et.al [21] used another type of pulse current for removal of organic matter from wastewater. However, his report didn't present any numerical data on voltage and time of changing electrode.

Effect of initial oil or milk concentration on turbidity removal efficiency

In Table 2- 4, theelectrical energy consumption (EEC) and time of reaction development and water recovery after EC versus initial oil or milk concentration during experiments are shown.

The effect of supporting electrolyte type and electrolyte dosage

Two tasks investigated, include

1) Access to maximum turbidity removal efficiency

2) A study on water recovery (or treated water) reuse. If the TDS of recovered water is near2 mS/cm or less, recycling is considered

economically feasible. In our study, electrolyte consumption to enhance EC performance is optimized in the range of 0.1-3.9 mS/cm for low to high concentration of oil or milk, and other operational parameters are reported based on these TDS levels (Table 1-4). However in the literature [3, 10, 14, 15, 16, 21] TDS levels is reported of up to 17 mS/cm. Thus electrolyte consumption at the highest concentration (10000 ppm) was measured as 0.86gr/L, whereas in the literature values up to 17gr/L are reported.

It should be noted that the turbidity removal efficiency in this study was 99.9%, the same value as reported in previous works.

In order to limit the IR-drop or solution resistance potential (η_R), conductivity of the solution should be sufficient. Most of the other works have used chloride as anion to enhance the conductivity of the solution and some have utilized nitrate and sulphate as the electrolyte [15, 16, 20]. The salt is employed to increase the conductivity of wastewater to be treated. The conductivity of the solution influences the current density, voltage and power consumption in EC process.

In this investigation,NaCl, KCl, PAC (poly aluminum chloride, Al_2 (OH)_nCl_{6-n}), NaNO₃ were used as supporting electrolytes separated by Al/Al electrodes. The performance of each electrolyte was compared in each batch, based on high turbidity removal efficiency and lowertime and energy consumption.

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NaCl and KCl have additional advantages because chloride ions can reduce the adverse effects of other anions such as HCO_3^{-1} and SO_4^{-2} [15]. The existence of the carbonate ion will lead to the precipitation of Ca^{2+} ion that forms an insulating layer on the surface of the electrodes. When polarization occurs, it will enhance the ohmic resistance of the EC cell. As a result, the conductivity of solutions are adjusted using supporting electrolyte in the range of 0.5-0.9Kg/m³ of wastewater based on initial concentration and type of solution.

Although turbidity removal increased using any supporting electrolyte types (Figure 11). The highest removal was observed with NaCl and KCl. As expected, the rate of organic matter degradation increased as the supporting electrolyte concentration increases.

At lower electrolyte concentration, the

turbidity removal is low compared to higher electrolyte concentration. For example, the removal efficiency of organic matter is near 99% at 0.9 Kg KCl /m³ wastewater, and 70% at 0.3 Kg KCl /m³.

In addition, energy consumption decreased as concentration of supporting electrolyte increased, because under constant current density the potential decreased. Energy consumption and turbidity removal efficiency were calculated by Eq (4) and (5) respectively. The optimized parameters are presented in Table 2-4.

Effect of PAC dosage on turbidity removal efficiency was also evaluated. The results showed that for removal of turbidity, PAC is not recommended. Also the influence of NaNO₃ on removal efficiency was examined. As Figure11 demonstrates, NaNO₃ and PAC



Figure 11. Influence of supporting electrolytes. EEC and time of reaction development and % Turbidity removal efficiency versus 5 types of supporting electrolyte in direct current (initial oil concentration, 1000 ppm oily waste water during experiments).

are not as good electrolytes as NaCl and KCl, based on the defined dosages. It is concluded that KCl is as effective as NaCl for complete removal of turbidity in these experiments, because of high removal efficiency and minimum energy consumption.

PAC as a coagulant aid combined with NaCl was used in these experiments to increase the removal efficiency and were compared with NaCl alone. Also the influence of NaCl + NaNO₃ was examined. Five types of electrolytes are compared in Figure 11. The electrical consumption; operating time and removal efficiency are shown versus supporting electrolytes.

Conclusion

The results of this study revealed that electrocoagulation (with Al/Al electrodes) can be applied for the treatment of industrial organic effluents containing oil or milk. Following conclusions are obtained:

(i) The optimum operating conditions in solutions containing initial (oil or milk) concentrations in the range of 1000-10000 ppm were obtained to achieve highest removal capacity (99%). The best optimized conditions were proposed are as follows:

Optimized range of electrical energy consumption (EEC): 2.3-12.15 kwh/m³ waste water, Optimized range of current density: 90-194.5 A/m², operating EC time: 7-22min, applied pH: 3-9 (optimum pH = 7 for oily waste waters and optimum pH = 5 for milky waste waters), applied voltage: 20 volts, (all of these data ranges are proposed based on initial concentration and type of solution).

(ii) After producing sludge, the passivity can interfere in EC and can control the mass transport. To overcome this problem, "alternating pulse current" (APC) and "pulse current" (PC) have been established to limit these resistances. Equal turbidity removal efficiency was obtained in direct current and alternating pulse current and pulse current. While in the PC mode, reaction time and therefore energy consumption is the lowest. In the APC and PC modes, the "water recovery" was significant, measuring as high as 0.92m³/m³ wastewater. Turbidity of recovered water in APC, PC and DC mode are under 5 and 10 and 20 NTU respectively after EC process. As a result, after APC or PC, the recycling of treated water is economically feasible. This is the first time that PC and APC are used for removing turbidity.

(iii) The impact of NaCl,KCl, PAC and NaNO₃ as supporting electrolyte and coagulant aid were evaluated. KCl is as effective as NaCl, and can remove organic matter from waste waters with the best performance in every aspect. The application of NaCl+PAC was ranked second, while NaNO₃+ NaCl was

t_{EC}

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third, based on removal turbidity and energy consumption. In this study, the electrolyte consumption for enhancement of EC is optimized in the range of 0.1-3.9 mS/cm for low to high concentration of milk or oil. It should be noted that the turbidity removal efficiency in this study was 99.9% the same as in previous works (that consume high electrolyte).

(iiii) Mixing (120 rpm) imposes significant effect on electrocoagulation time, actually 50% reduction in time and energy consumption, is observed.

(iiiii) The optimized parameters enhances the treatment rate to reduce oil or milk concentrations below admissible legal levels (turbidity under 5 NTU, COD output < 200 mg/L).

Nomenclature

- APC Alternating pulse current(or alternating square wave)
- C_0 Initial oil concentration, (ppm)
- CD Current density (A/m² of anode)
- EC Electrocoagulation
- EEC Electrical Energy consumption, (Kilo watt hour/m³ waste water)-or power consumption.
- I Operating amperage (ampere)
- NTU Nephelometer turbidity unit
- PAC Poly aluminum chloride
- PC Pulse current (or square wave)

ppm Part per millions(this unit in water science is very common for concentration)

 $(1ppm = 1mg / lit = 10^{-3} \times kg / m^3)$

- Time of electrocoagulation or reaction, min,
- V Operating voltage(volt)
 - Volume of wastewater (m³).
- γ Specific Conductivity, mS/cm (millisiemense per centimeter). the SI unit of conductance is called siemens, S, has A² sec³/(Kg.m²) dimension

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