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Original Article

Testing the Removal of Microplastics from Surface Water Using Okra Mucilage Combined with Polyaluminium Chloride (PAC)

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Abstract: Recently, microplastics (MPs) have become an emerging pollutant. Coagulation-flocculation is a physicochemical method that uses chemical coagulants to remove MPs, but the disadvantages include high cost and chemical residues that are harmful to the environment. Okra mucilage is a low-cost natural coagulant that has been found to be capable of removing MPs from aquatic environments. The objective of this study was to test the environmentally friendly combination of okra mucilage with Polyaluminium chloride (PAC) to remove MPs from surface water. The results showed that okra mucilage did not change the pH of the water and was suitable for the optimal pH range of PAC. The ratio of okra mucilage to PAC was 2:1 (mL:mg) per liter of water. The optimal coagulation time to remove microplastics in surface water was 15 min with a removal efficiency of about 62.96% of MPs. However, depending on the state of the floc/coagulant, whether it is settling or floating, we still need to develop an optimal method to separate the floc/coagulant from the water environment.

Keywords: Microplastics, Okra mucilage, Polyaluminum chloride (PAC), removal, surface water.

1. Introduction

Population growth, urbanization, and industrialization have led to increased demand for plastics [1], and plastics pollute many environments [2]. When plastics degrade, they become brittle and break into smaller fragments and particles [3], forming microplastics (MPs) [4]. MPs originating from primary and secondary sources [5, 6] have been found in all

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hemispheres of the world [7]. MPs are one of the emerging pollutants [8] and have caused much concern [9], affecting the food chain and wildlife health [10].

There are many methods for removing MPs with different mechanisms and efficiencies [11], and they need to be environmentally friendly [12]. Coagulation-flocculation is widely used to remove suspended or colloidal particles in water [13] with chemical coagulants to destabilize colloids and suspended solids [14]. However, chemical coagulants have disadvantages such as high cost, potential environmental damage and human health concerns [15, 16]. Natural coagulants for water treatment have attracted attention in developing countries [17], due to their non-toxicity, biodegradability, stable shear strength, easy accessibility and low cost [18].

In addition, researchers have found that okra mucilage has the potential to remove MPs from aqueous media [19] due to its low sludge formation and safety [20]. Okra mucilage has properties such as water absorption, oil absorption, emulsification, foaming, and complexation [21]. Meanwhile, MPs have hydrophobic properties [22] and adsorption capacity [23], which makes them capable of carrying certain contaminants. On the other hand, okra mucilage has been studied as a natural coagulant in coagulation-flocculation process for the treatment of industrial textile wastewater [14].

Therefore, there is a real need to develop new environmentally friendly methods for removing MPs from water. In this context, this study evaluated okra mucilage as a biocoagulant when combined with PAC for the treatment of MP contaminated water.

2. Materials and Methods

i) Preparation of PAC chemical: Powdered PAC (India), with the molecular formula $[Al_2(OH)_nCl_{6-n}]_m$, is an alum with an Al_2O_3 content of 28 - 30%, alkalinity from 40 - 90% and a potential pH range of 5 - 8.5 [24];

ii) Preparation of microplastic source: Recently, reports have shown that MPs are present in some samples of domestic wastewater in large urban areas such as Hanoi [25], Binh Duong [26] or urban lake in Da Nang [27] or rivers flowing through densely populated and urbanized urban areas such as To Lich, Day and Nhue [28]. Currently, the domestic wastewater discharges into the Bun Xang Lake have seriously polluted the surface water quality [29] and due to the long-term uncollected waste, it leads to pollution [30]. Therefore, Bung Xang Lake (located around 3 wards Xuan Khanh, An Khanh and Hung Loi in Ninh Kieu district) was chosen as one of 63 unfilled lakes, canals and ditches in Can Tho city [31] to collect MPs;

MPs in Bung Xang Lake surface water were sampled at the coordinates 10°02'01"N, 105°45'52"E using an Albatross 7 sampler (Pirika, Japan) attached to a motor (12 V rechargeable battery) with a propeller that creates a sliding flow at a stable speed, pushing the water inside the device with a sampling net (250 µm mesh), MPs are retained inside the sampling net, the water flows out of the net for 60 min at a low Bung Xang Lake water level. The sampling net brought back to the laboratory was hung high and washed on the outside with tap water (a tap with a mesh cover with 250 µm holes to avoid contamination of the MP sample from the outside), then washed inside the net so that the MP particles flow to the bottom of the net using a sampling funnel. Next, the water sample was filtered through a 1 mm sieve to remove trash mixed in the sample. The water sample passed through the sieve 1 mm was then filtered through a 250 µm sieve. The MP particles on the 250 µm sieve were transferred to a 250 µm mesh and dried at 50 °C in a drying oven (Memmert UN110, Germany). The dried MP sample was soaked in 30% H_2O_2 solution (Merck, Germany) for 24 h to remove organic compounds. Then, the sample was filtered through a 250 µm sieve, washed with distilled water (filtered through GF/C filter paper, Whatman - UK) and transferred to a 250 µm mesh and dried at 50 °C in a drying oven. Finally, the obtained MP sample was purified from impurities;

iii) Procedure to prepare Okra mucilage: Fresh okra is collected from the market, washed with clean water, cut into 2 - 4 cm pieces, added distilled water (ratio of Okra: water = 1:2, kg/L) and heated at 90 °C, stable for 60 min, then cooled to room temperature. Finally, filter the solution through sieve (mesh 1 mm) to remove the Okra residue (filter through 1 mm mesh sieve). According to Kalkan & Maskan (2023), the extraction temperature of okra mucilage was 25-80 °C, the extraction time was 1-7 h, but the optimum extraction time was 1.52 h and the water to raw material ratio was 4:1 – 10:1. Okra mucilage had an emulsifying capacity of 47.05% and reduced the oil content of the mixture by 95.4% [32];

iv) Procedure to prepare hypothetical water samples (microplastic pollution): Surface water samples were collected at Bung Xang Lake at the same location as MP sampling, water samples were collected 20 cm below the water surface in a 30 L plastic can with a total sample volume 120 L. At the laboratory, water is filtered through a 1 mm sieve to remove trash, then through a 250 µm sieve to remove initial MPs. Finally, stir the water well and divide it into 2 parts: 30 liters (WP1) without adding MPs for perform experiment 2.1 and 90 liters (WP2) have adding purified MPs from section ii and mix well to create a hypothetical surface water source completely contaminated with MPs to perform experiment 2.2;

The process of removing MP from the water environment is carried out in 2 steps: Step 1, check the change in pH and turbidity when increasing the volume of Okra mucilage into the experimental water; the dosage of PAC and determine the appropriate ratio of Okra mucilage:PAC is carried out as in section 2.1. Step 2, test the ability to remove MPs of Okra mucilage combined with PAC on the Jartest device in the laboratory is carried out as in section 2.2.

2.1. Test the Change in pH, Turbidity and Ratio of Okra Mucilage:PAC

There are 3 experiments performed as follows:

i) Determine the change in pH, turbidity: Measure 1 L of WP1 water (prepared in *section iv*, this water represents Bung Xang Lake water, but has removed MPs) into a glass cup and stir well with a magnetic stirrer (Velp T.are, Italia) at a speed of 70 rpm, each experimental cup adds a volume of okra mucilage from 1 to 20 mL, then determine the change in pH (WTW Multi 3510 IDS, Germany) and turbidity (Lovibond TB211 IR, Germany) to find the appropriate pH range for PAC coagulant and volume of Okra mucilage;

ii) Determination of Okra mucilage volume: According to Zhang et al., (2022), the dosage of PAC combined with polysaccharide extracted from seaweed (*Enteromorpha prolifera*) was from 1 to 4 mg/L to remove organic substances [33]. In addition, when the initial turbidity of raw water was low, the dosage of PAC used was 1 mg/L at pH = 7 [34]. The dosage of PAC = 1 mg/L was selected to be performed on 1 L of WP1 water (see *section iv*) for each change in okra mucilage volume (from 1 to 20 mL) and coagulation time (10, 15, 20, 30, 45 and 60 min) through measuring the turbidity of the water (Lovibond TB211IR, Germany);

iii) Determine PAC dosage: From the volume of Okra mucilage determined in step 2.1b when they give the lowest residual turbidity. Perform on 1 L of WP1 water (see *section iv*) for each change of PAC dosage from 1 to 4 mg/L with flocculation settling time (10, 15, 20, 30, 45 and 60 min) through measuring the residual turbidity to find the appropriate PAC concentration and Okra mucilage:PAC ratio and flocculation settling times.

2.2. Testing the Ability Removal of Microplastics of Okra Mucilage Combined with PAC

i) Determine the MPs concentration: Stir WP2 water (*see section iv*) well, measure 1 L and filter through a 250 µm sieve, wash the sieve with distilled water (filtered through GF/C filter paper) and transfer the washing water containing MPs on the sieve into a beaker. The procedure for extracting MPs from surface water according to Mekong River Commission guidelines (2024) is as follows: add 50 mL solution H_2O_2 (30%, Merck, Germany) and 50 mL solution $FeSO_4.7H_2O$ (Merck, Germany: weigh 7.5 g dissolved in 3 mL H_2SO_4 concentrated, diluted in a 500-mL flask with water) and cover with aluminium foil to remove and dissolve the organic matter that is attached to the surfaces of MPs (Fenton reaction). After 72 h, filter through a 250 µm sieve, wash and collect the washing water containing MP particles on the sieve into a beaker. Finally, filter the MPs through GF/C filter paper (Whatman, England) on a vacuum filtration system (Schott Duran, Germany) [35]. Determine the morphology and count the number of MPs using a microscope (Nikon Eclipse CI-L plus, Japan) with 40× magnification. MP counts are defined as the total number of all forms such as particles, fibers, films and fragments;

ii) Experiment on Jartest system: From the ratio parameter of Okra mucilage:PAC determined in step 2.1 (b, c). Conduct the experiment simultaneously on the Jartest system (Daihan JT-M6C, Taiwan) with 6 beakers, each beaker containing 1 L of WP2 water (*see section iv*). First, add the volume of Okra mucilage determined from step 2.1b, turn on the Jartest device to mix the sample well. Then add the dosage of PAC determined in section 2.1c. Stir quickly at 150 rpm for 1 min, stir slowly at 50 rpm for 15 min. Turn off and pull up the stirring blade of the Jartest device. Monitor the flocculation/coagulation process to see if it is settling or/and floating and take measures to separate them from the water environment over time (10, 15, 20, 30, 45 and 60 min). Take the flocculated/coagulated (settling and/or floating) residue to determine MP according to the Mekong River Commission analysis procedure (2024) [35] and observe the morphology and count the quantity as in step 2.2a. The MP removal efficiency is calculated according to formula (1)

$$\text{Efficiency removal (\%)} = \frac{\text{Number of MPs attached to coagulated flocs}}{\text{Number of input MPs}} \times 100 \quad (1)$$

3. Results and Discussions

3.1. Change in pH, Turbidity and Ratio of Okra Mucilage:PAC

i) pH, turbidity

The results showed that Okra mucilage did not change the pH of the water, the initial pH of the water was 7.15, the pH change did not differ between the volumes of Okra mucilage added, around the value 7, until the volume of Okra mucilage added reached the highest (20 mL), the pH reached 7.23, not significantly different from the initial value. According to Nti et al.; (2021) the optimal pH range for PAC coagulation efficiency is from 7.5 to 8.0 [36], which shows that Okra mucilage does not affect the activity of PAC;

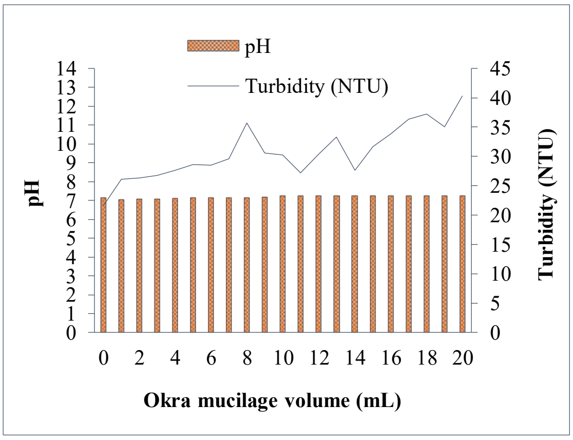

Figure 1. Change in pH, turbidity according to Okra mucilage volume.

In contrast, as the volume of Okra mucilage increased, the turbidity of the water tended to increase, but there was a fluctuation between the volumes of Okra mucilage added. Specifically, the turbidity increased linearly with the volume of Okra mucilage from 1 to 8 mL. From the volume of Okra mucilage from 8 to 14 mL, the turbidity fluctuated significantly. This trend also appeared until the volume of Okra mucilage added reached 20 mL. The results showed that when adding Okra mucilage volume to 20 mL, the maximum raw water turbidity reached 40.3 NTU. According to the recommendation of [25], water turbidity from 15 - 90 NTU, the dosage of

PAC needed to use is 6-10 mg/L. However, PAC is very effective at low doses, if overdosed PAC will dissolve colloidal particles [24]. In addition, PAC combined with polysaccharide extracted from seaweed to remove organic substances has a dosage of 1 to 4 mg/L [33] and when the initial turbidity of raw water is low, the PAC dosage used is 1 mg/L at pH = 7 [34];

Thus, Okra mucilage does not change the pH of raw water and the optimal pH range of PAC. The appropriate PAC dosage is from 1 - 4 mg/L when the turbidity of raw water is low (40.3 NTU);

ii) Okra mucilage volume

When changing the volume of Okra mucilage from 1 - 20 mL, while fixing the dosage of PAC (1 mg/L), it showed that the residual turbidity in the volume of Okra mucilage of 2 mL, 3 mL and 4 mL was lower than the other volumes of Okra mucilage for all settling times. In which, the residual turbidity at the volume of Okra mucilage of 2 mL had the lowest (Figure 2). Thus, the okra mucilage volume of 2 mL is more optimal than other volumes as they give the smallest residual turbidity;

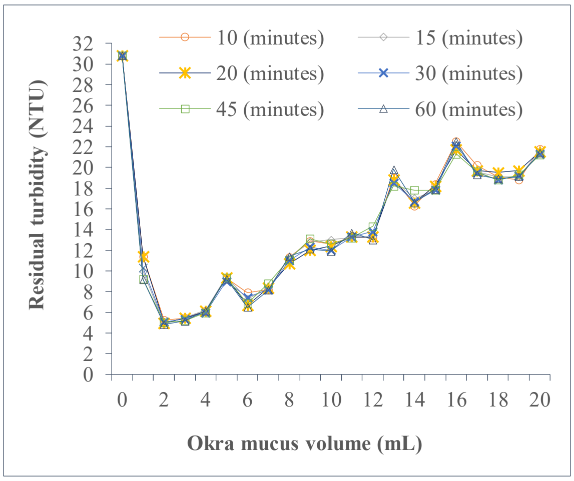

Figure 2. Change in Okra mucilage volume (fixed PAC dosage = 1 mg/L).

iii) PAC dosage

The optimal Okra mucilage volume (2 mL), change the PAC dosage (1; 1.5; 2; 2.5; 3; 3.5 and 4 mg/L), the results showed that the residual turbidity at 1 mg/L PAC dosage was the lowest in all flocculation settling times (Figure 3);

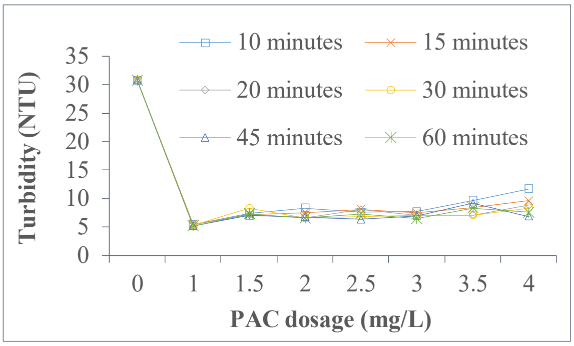

Figure 3. Change in dosage of PAC combined with 2 mL of Okra mucilage.

As the PAC dosage increased to greater than 1 mg/L, the residual turbidity began to increase at all Okra mucilage volumes and all flocculation settling times. Although PAC is effective in coagulating organic matter and precipitating flocculant compounds and suspended substances, excessive dosage will cause re-stabilization of colloidal particles. Therefore, the residual turbidity increases again when the PAC dosage increases. Thus, the PAC dosage is highly effective at 1 mg/L compared to other dosages.

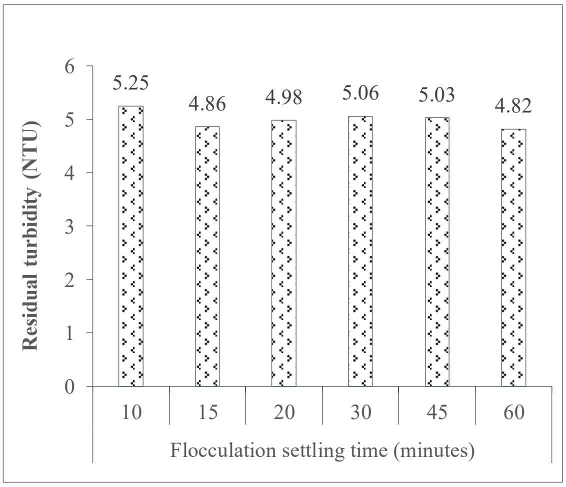

Figure 4. Flocculation settling times.

From the results in Figure 4, the residual turbidity at 60 min of flocculation time was the smallest (4.82 NTU), followed by 15 min of flocculation time with 4.86 NTU. However, the flocculation time had to be extended by 45 min, but the difference in turbidity between 15 min and 60 min of flocculation time was not significant (4.86 NTU vs. 4.82 NTU). Therefore, choosing 15 min of flocculation time was more effective than 60 min of flocculation time.

Thus, from the results, the parameters for operating the Jartest process with the volume of Okra mucilage is 2 mL, the dosage of PAC is 1 mg/L (ratio 2:1), fast stirring time 150 rpm for 1 min, slow stirring at 50 rpm for 15 min and settling time 15 min.

3.2. Microplastic Removal Ability of Okra Mucilage Combined with PAC

i) Initial MPs concentration

The total initial number of MPs in 1 liter of raw water reached 135 ± 10.13 items/L, with the main shapes being fragments, fibers and particles as Figure 5. Compared with the actual density of MPs polluting surface water in Da Nang inner-city lakes from 0.85 particles/L to 1.5 particles/L [27], the surface water assumed to be contaminated with MPs in this study has a high concentration of MPs to increase the chance of MP adhesion of Okra mucilage and PAC during the coagulation/coagulation process;

(a) (b) (c)

Figure 5. Microplastic shapes: (a) fragments, (b) fibers, and (c) particles.

ii) Microplastic removal efficiency

In this experiment, MPs input was 135 particles/L, with Okra mucilage volume of 2 mL and PAC dosage of 1 mg/L, fast stirring time of 150 rpm for 1 min, slow stirring of 50 rpm for 15 min with settling times of 10, 15, 20, 30 45, 60 min. Okra mucilage could absorb water, absorb oil and dissolve in water, helping Okra mucilage and suspended substances in water to come into contact easily. In addition, Okra mucilage also could emulsify foam and form complexes [37]. When Okra mucilage is added to raw water (before PAC is added), this causes MPs to adhere to the Okra mucilage. Okra mucilage mixtures contain hydrocolloids, which form gels in the presence of water composed of various carbohydrates such as alginates, showing potential in their divalent cationic cross-linked form as adhesives [38]. Then, PAC is added, it coagulates Okra mucilage, organic substances and precipitates flocculent compounds and suspended substances. The results show that the flocculation/coagulation process changes over different times. They divide into two parts, most of them settle and one-part floats to the top as shown in Figure 6;

Okra mucilage contains polysaccharides which are used in wastewater treatment fields [39], they are chemically inert, non-toxic, biodegradable and biocompatible [40]. Polysaccharides contain polyhydroxy groups hence they are water soluble and hence used as hydrophilic natural polymers [41]. The anionic nature of okra polysaccharides promotes flocculation of heavy metals from water [42].

Results from studies have shown that Okra mucilage acts as a natural coagulant aid because it is water soluble and can destabilize colloidal particles, flocculate small particles [19] and increase flocculation and sedimentation efficiency, reduce the amount of coagulant [20] with increased floc size and short time to reach maximum floc size, high particle adsorption capacity [21].

Extraction the floating floc and flocculation floc (Figure 6) for MP analysis showed that the results of the number of MP particles attached to the floating flocs at 15 min had the smallest number of MP particles (29 items) compared to the times 20, 30, 45 and 60 min, respectively 36, 42, 63 and 66 items (Table 1). In contrast, the MPs particles attached to the flocculation flocs at 15 min have the highest number of particles (56 items), after this time, the number of MP particles decreases until 60 min (Table 1). The disadvantage of PAC is that it easily causes destabilization of colloidal particles, due to prolonged time, causing coagulation/flocculation flocs to break and float again. This means that the number of MPs in the floating aggregates is significantly more than at the 15 min time point.

Thus, at each flocculation settling time, the flocculation/coagulation flocs have two trends including settling and floating. This is an important factor to have appropriate measures to separate flocculation/coagulation flocs from water.

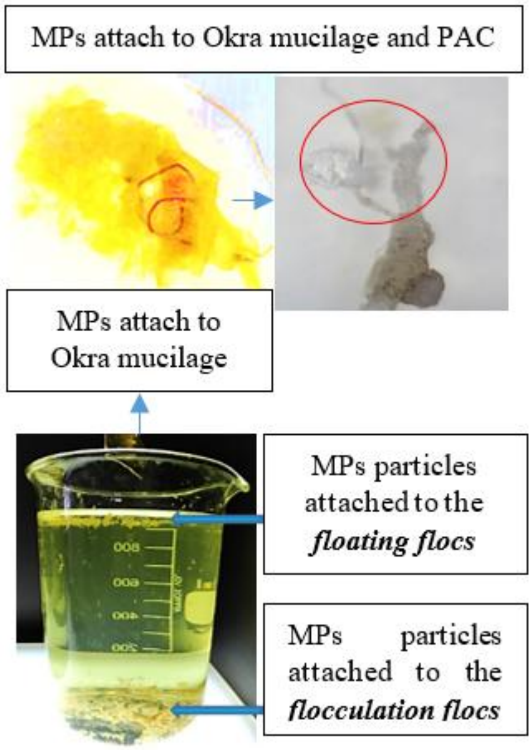

Figure 6. Flocculation/coagulation process of Okra mucilage combined with PAC and MPs image under a 40× magnification microscope.

Table 1. Efficiency removal

Settling time (*minutes*)	**10**	**15**	**20**	**30**	**45**	**60**
Number of input MPs (*items*)	135	135	135	135	135	135
Number of MPs particles attached to the floating flocs (*items*)	38	29	36	42	63	66
Number of MPs particles attached to the flocculation flocs (*items*)	40	56	23	14	16	20
Efficiency removal (Floating flocs) (*%*)	28.15	21.48	26.67	31.11	46.67	48.89
Efficiency removal (Flocculation settles) (*%*)	29.63	41.48	17.04	10.37	11.85	14.81
Total efficiency removal (*%*)	57.78	62.96	43.70	41.48	58.52	63.70

Total ffective removal of MPs in each flocculation settling time (including flotation in and settling in Table 1) showed that at 10 min it reached 57.78%, at 15 min reached 62.96%, at 20 min reached 43.70%, at 30 min reached 41.48%, at 45 min reached 58.52% and at 60 min reached 63.70%. Thus, the results show that 60 min is the most effective, followed by 15 min. However, the effective removal difference between 60 min and 15 min of flocculation settling time was 0.74%, but an additional 45 min was spent. Therefore, choosing a flocculation settling time of 15 min is more effective.

The hydroxyl groups of okra mucilage act as active sites for the removal of colloidal particles, turbidity in water treatment as they replace inorganic coagulants by about 64% [22]. Other studies have shown that okra is effective in removing turbidity at much lower concentrations. The polymers in okra mucilage exhibit high flocculating capacity at low dosages without any pH change [43], which can remove 95% of suspended solids and 65% of dissolved solids from wastewater. After 1 h, maximum solids removal can be achieved with an Okra mucilage concentration of 0.04 mg/L [44].

4. Conclusion

Okra mucilage combined with PAC to remove MPs from water is about 62.96% effective. However, depending on the state of the flocculation/coagulation floc that settles or floats, there are measures to effectively separate them from the water. Okra mucilage does not change the pH of water and is suitable for the optimal pH range of PAC. The ratio between Okra mucilage and PAC dosage was 2:1 (mL:mg) per liter with a flocculation settling time of 15 min.

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