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Determination of Active Constituents of Some Household Cleaners Available in Benghazi Markets

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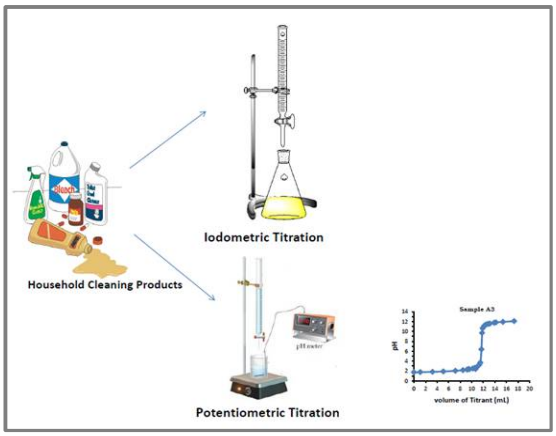
ABSTRACT

In the present study a total of 28 samples of different domestic cleaning products were randomly purchased from Benghazi markets-Libya to detect the active components concentrations and the pH values. The obtained data were compared with the respected product`s label and Libyan Standards Limits. The pH values of the cleaning products were measured using pH-meter connected with glass combined electrode. The products were analyzed using iodometric method, to determine the concentration of sodium hypochlorite in bleach samples. Potentiomtric titration was used to determine total acidity% of anti-scale cleaners and alkalinity% of drain open and kitchen cleaners. The results of analysis showed that there were 5 bleaching samples had sodium hypochlorite concentration above the limit set by Libyan Standard Legislation (5%) and all the anti-scale cleaners had concentration of hydrochloric acid below the limit set by Libyan Standard. The total alkalinity% of the open drain cleaners were in agreement with the concentrations available on products package label.

Graphical Abstract



Titrimetric Methods for Analysis of some Household Cleaners.

Keywords: Household cleaning product, Bleach products, Sodium hypochlorite, Total acidity%.

INTRODUCTION

Household cleaning products like other consuming products are sold in large quantities around the world, and consequently the main chemical ingredients in these products are often high production volume chemicals [1], these chemicals are responsible to human health-related defects [2, 3]. A cleaning product is defined as any material used for cleaning or disinfecting the surfaces in general work environments. These products have become an indispensable part of modern life, as they are used on daily basis in nearly all workplaces and homes. Different types of cleaning agents have been produced to help on dust and dirt removal, and for disinfection and surface maintenance [1]. Household cleaning products composed of active components that has essential role of action of cleaner, and other components. Therefore, these cleaners are generally classified according to the application and action of major components of cleaning products include disinfectants, detergents, solvents, alkaline agents, acids, complexing agents, corrosion inhibitors, preservatives, polishes antimicrobial compounds, abrasives and bleaching agents[3, 2].

Household bleach is a strong and effective disinfectant. The active ingredient of bleach cleaner is sodium hypochlorite (NaOCl) and chlorine gas (Cl_2). The sodium hypochlorite is a weak base that denatures protein in micro-organisms and is therefore effective in killing bacteria, fungus and viruses. Diluted household bleach is recommended for the disinfection of facilities. However, sodium hypochlorite is classified as corrosive substance and should be labelled with "Hazard Statement", it causes severe skin burns and eye damage. These characteristics must be kept in mind during transport, storage and use of sodium hypochlorite [1, 4]. In fact, there are some places in home where really tough cleaning jobs. For these jobs, cleaners are formulated with extremes in pH, which allow the acidity or alkalinity of the cleaner to quickly attack the un-wanted dirt, grease or stain. The acidic household cleaners usually contain hydrochloric acid, phosphoric acid, sodium bisulfate, or hydroxyacetic acid, which remove alkaline scale. In the other hand, the basic cleaners usually contain alkaline agents, such as, ammonia, sodium hydroxide, sodium carbonate and silicates. The weaker bases cut grease, while the strong bases dissolve animal matter such as hair, grease, and foodstuffs. The composition of drain open cleaner consists of alkali metal hydroxide which is either sodium hydroxide or potassium hydroxide. Furthermore the composition of these products may also include an acidic agent such as sodium bisulphate, citric acid and an oxidizing agent such as, sodium carbonate, peroxide, sodium perborate monohydrate or peroxymonosulphate. Optionally, an anionic or nonionic surfactant foaming agents can be included [1, 2].

The bleach and extremes pH cleaning products are heavily corrosive materials. On Contact, these products have ability to irritate the eyes, skin, mucous membranes and respiratory tract often by inhaling the emitted gases, during the use of cleaning products. Other effects included; emphysema, respiratory diseases, heart diseases and cancer may show up years after exposure. Such cleaners should be handled with extreme caution and thick rubber gloves should be worn when using them [1, 2]. As the cleaning products are disposed of in regular garbage, in waste water or by dumping onto the ground, the chemical components of these products may act as pollutants in home and also contribute to environmental pollution [2]. In USA, the Environmental Protection Agency (USA-EPA) classified the residue of domestic use, house care products, pest control and garden and automotive maintenance and medicine, as contaminants [5, 6]. Similarly, other authors classified as contaminant waste, those residues generated from using cleaning products, pesticides, paints, varnish etc, produce in small amounts in houses and that may cause serious health problems due to their toxic, reactive, corrosive and flammable characteristics [6].

Many recent studies have focused on different types of cleaning products as its widely used and plays a major role in our daily life. Although there were many researches on the adverse effects of the continuous exposure to domestic cleaners, only few researches have taken the amount of active ingredients in consideration. However, numerous methods were published for quantitative analysis of

the active components of some cleaning products, including bleaching (sodium hypochlorite) toilet (acid), drain and kitchen (base) cleaners.

To determine the concentration of sodium hypochlorite in the most common commercial household bleach products, Sitanurak*et al*., have fabricated membraneless gas-separation microfluidic paper-based analytical device [7]. Also, a fluorescence detection technique, based on bovine serum albumin stabilized gold nanoclusters, was developed to detect trace levels of hypochlorite ion in drinking water samples [8]. Different titrimetric and colorimetric methods have been successfully applied to determine the hypochlorite ion in various samples of natural water, tap water and milk [9-11]. An anodic cyclic voltammetric method using different electrodes was optimized to determine free chlorine and hypochlorite ion contents in disinfection and bleaching solutions [12, 13]. Several authors were determined the hypochlorite ion and free available chlorine contents, in irrigation solutions, that mostly used in endodontic treatments, and commercial bleach solutions using iodometric method [14-19].

The measurements of pH values and determination of total acidity and total alkalinity are the most important factors in determining the cleaning quality of household cleaning products. The acidic (or basic) components in the cleaning products are usually determined by potentiometric titration using a glass electrode and pH meter [19, 20]. Titrations are often recorded on titration curves, whose composition are generally known. The equivalent point of the titration obtains when pH of the reaction is just about to 7 [20, 21]. Even more, potentiometric titration in presence of different types of ion selective electrodes have been used to determine ionic and non-ionic surfactants [22, 23].

However, several authors expressed the total acidity and total alkalinity of some cleaners as titratable acid reserve (TAR), and similarly titratable alkaline reserve (TAR), respectively [24-26]. Hoffman was defined TAR as the number of millliters (mL) of 0.1 M solution of hydrochloric acid (or sodium hydroxide) required to titrate 100 mL of 1% solution of test cleaning product to pH 8.00. Hoffman *et al*., mentioned that the pH value of 8.00 was chosen to approximate normal esophageal pH [24]. Navratil *et al*., has defined TAR for some anti-scale cleaners as the amount of sodium hydroxide necessary for achievement of chosen pH in 100 mL of lime-scale remover [25].

The aim of the current study is evaluating the pH values and sodium hypochlorite concentration, with the corresponding free available chlorine contents in some commercially household bleaches samples using iodometric method. Also the total acidity and total alkalinity contents in some anti-scale (toilet bowl) cleaners, kitchen and drain open cleaners, respectively, have been determine using potentiometric titration method. All the household cleaning products were collected from the local markets in Benghazi city. Our results will be compared with the concentration of active ingredients on available label and the National Libyan Standards Regulation for the active ingredients in the investigated household cleaning products.

MATERIALS AND METHODS

Chemicals and Equipments: All of the chemicals and reagents used were of analytical grade. These chemicals included, glacial acetic acid (CODEX), sodium thiosulphate, potassium iodide and soluble starch (BDH), potassium iodate (Riedel-deHaen), sulphuric acid (98%, Hopkin& Williams Chemical Ltd), hydrochloric acid (Merck), sodium hydroxide (Riedel-deHaen). pH-meter(*Ino lab WTW*) equipped with glass combined electrode (*pH-electrode senTix*61-*B*023009*AP*017) were used to measure pH values of the household cleaning solutions [27, 28]. A clean and dry density bottle (pycnometer-25 mL) is used for density measurements [29].

Sampling: In this study, twenty eight household cleaning samples, included twenty bleach cleaning products and five anti-scale cleaners, one kitchen cleaner and two drain open cleaners, were collected

randomly by purchasing them from different cleaning products retailer in various regions of Benghazi-Libya. All data of the household cleaning products are shown in table 1 and table 2.

Table 1. List of Household Bleach Cleaning Products

Sample code	Product name	Manufacture company	Ingredient
B1	Clorol[a]	Egypt	NaOCl<5%, NaOH< 5%, H_2O>30%
B2	Clorox[a]	Egypt	NaOCl<5%, NaOH< 5%, soft H_2O > 30%
B3	Al-Shallal[a]	-	NaOCl<5% NaOH< 5%, H_2O>30%
B4	Lotus	Libya	-
B5	Al-Arij	Libya	-
B6	Al-Rakey	Libya	-
*B7-B20		Libya	-

*Sample B7- B20 were collected from different local factories in Benghazi. These Samples have no package container label. [a]Manufacture & Expiry Dates of these products have mentioned.

Table 2. List of Anti-scale, Kitchen and Drain Open Cleaning Products

Sample code	Product name	Manufacture company	Ingredient
A1	Vixal[a]	Indonesia	17% HCl
A2	HARPI[a]	UK	Benzyldimethltridecyl-azoniumchloride
A3	FLASH[a]	Turkey	HCl
A4	Good Maid[a]	EU	Aqua,7-9% HNO_3,1-3% H_3PO_3
A5	T-Top	Libya	-
K1	CETRIC[a]	Egypt	Nonionic active matter (≤5%), NaOH
D1	Kalyon[a]	Turkey	100% NaOH
D2	ERFRES[a]	Turkey	>99% NaOH

A1-A5: anti-scale cleaners, K1: Kitchen cleaner, D1and D2 Drain Open cleaners.
[a]Manufacture & Expiry Dates of all these products have mentioned, except Sample A5.

Determination of Sodium hypochlorite in Bleach Cleaning Products: Sodium hypochlorite content of bleach cleaning products is determined by iodometric method. In this method, 10 mL of bleaching sample solution was transferred to a 250 mL stopper conical flask. Then 3 mL of potassium iodide solution was added, followed by 2 mL glacial acetic acid. The liberated iodine was titrated with standard 0.1000 M sodium thiosulphate solution. When the color has faded to a pale yellow, about 2 mL of starch solution was added and the titration was continued drop by drop until the solution just become colorless. The volume of titration was recorded and the percentage by weight of sodium hypochlorite and the corresponding active chlorine, in the bleach cleaning product, were calculated using equations 1 and equation 2, respectively. The titration process was repeated with other two 10mL of the sample solution [**15, 20**].

$$\text{sodium hypochlorite (w/w\%)} = \frac{\text{weight of NaOCl}}{\text{sample weight}} \times 100 \qquad \text{Eq. 1}$$

$$\text{available chlorine (w/w\%)} = \frac{\text{weight percent of NaOCl}}{1.05} \qquad \text{Eq. 2}$$

Potentiometric Titration of Household Cleaners: The total acidity of household cleaning products was determined by potentiometric titration method using standard sodium hydroxide solution. 10 mL of sample solution is quantitatively transferred to a 100 mL volumetric flask and filled to the mark with water. 10 mL of this solution was pipette into a 100 mL beaker. Then the combined glass electrode is inserted in the solution, the magnetic stirrer is adjusted and a burette containing 0.1000M sodium hydroxide solution is set up. The pH of the solution before the addition of any titrant is measured. Then, about 1 mL of the base solution from the burette is added and again the pH value of solution is measured. This step is preceded in the same manner to record the pH and burette readings after each addition.

The same procedure is used to determine the total alkalinity of kitchen and drain open cleaners, but in this case 0.1000 M hydrochloric acid is used as a titrant. The titration process is repeated three times using a new aliquot of cleaning product solution. The volume of neutralization is determined from the titration curve and then, used to calculate the concentration of the hydrochloric acid (or sodium hydroxide) in household cleaner, which expressed as total acidity% and total alkalinity% using equation (3) and equation (4), respectively [**20, 21, 25**].

$$\text{Total acidity \%} = \frac{\text{M} \times \text{V} \times \text{Molecular weight}}{\text{sample weight (g)} \times 1000} \times 100 \qquad \text{Eq. 3}$$

$$\text{Total alkalinity \%} = \frac{\text{M} \times \text{V} \times \text{Molecular weight}}{\text{sample weight (g)} \times 1000} \times 100 \qquad \text{Eq. 4}$$

Where; M = Molarity of titrant, V = volume of titrant

Statistical Analysis: The analysis of the household cleaning samples was performed in triplicate and the results were expressed as the mean values with standard deviation (mean ±SD) of w/w%. Statistical analysis of data was carried out using Statistical Package For Social Analysis (SPSS) (SPSS version 19.0, IBM, Chicago, III., USA) Program, adopting the significance level of 5% ($P< 0.05$). The mean values were Compared using dispersion analysis (ANOVA). For the multiple comparisons, a Least Significant Difference (LSD) test was applied. Significant difference between the mean values and the available specification label concentration were determined using One Sample t-Test.

RESULTS AND DISCUSSION

In the present study, twenty eight household cleaning products were randomly collected for chemical analysis. These samples included twenty bleach products, five anti-scale (toilet bowl) cleaners, one kitchen cleaner and two drain open cleaners. The pH values of the cleaning products were measured and percentage of active ingredients was determined using suitable analysis methods [**21-22, 27-28**]. As we know Labelling of cleaning products plays an important role in the prevention and treatment of product exposure. Since the label should be a source of toxicological information and using instructions. According to the Libyan National Standard Regulation, and the Material Safety Data Sheet (MSDS), the container of the cleaning products should be suitable, does not effect by the product, and tightly closed. Also the label on the package should show the name of the product and its trade mark, place of manufacture, validity date, all hazardous substances (the active ingredients) and their precise percentages, information concerning safe use, handling, and storage of product, instructions to first aid [**30-32**]. As shown in table 1, three bleach samples were imported (B1, B2 and B3). The label of these products specified sodium hypochlorite very imprecise (i.e. <5% sodium hypochlorite). Some of the selected samples sold by national distinct companies in Libya, such as sample B4, Sample B5 and sample B6, the labels on the packages of these bleaching products did not indicate the levels of sodium hypochlorite. The other bleach samples were non-disclosure the product label. They vended to the public in different quality and capacity containers. As shown in table 1, only the three imported bleach products were vended in opaque airtight containers with disclosure product label indicted the manufacture and expiry dates of their productions. Clarkson *etal*. pointed out the necessity of storing sodium hypochlorite inside closed opaque containers, because sodium hypochlorite solution deteriorate with time and temperature. Its stability is also reduced by exposure to light, which cause loss of chlorine content in the bleach solution [**17, 33**]. One bleaching product was turbid (Sample B15). This sample might prepared using hard tap water. The nature of precipitate in the hard water solution is not known, but may be calcium or magnesium salts precipitated by the high pH of bleach solutions [**17**].

All the selected acidic and basic cleaning products were imported from different countries, except sample A5, table 2. Unfortunately, those products use by house-wives. The labels of the vessel of sample A1 and sample A2 were written in foreign languages. Therefore, the information of using the

product and the warning instructions were not understandable by consumer. For sample A5, the information that only available in this sample package label is trade name of the product, table 2.

Measurement of pH values of Household Cleaning Products: The results recorded in table 3, show that the pH values of bleach samples were ranged between 8.10 and 12.94. The commercial sodium hypochlorite solution should be strong alkaline [17, 33].

Table 3. Physiochemical Properties of Household Cleaning Samples

Physiochemical properties				
Household Cleaners	pH		Density (g mL^{-1})	
	Mean ±sd	Range	Mean ±sd	Range
Bleach (B1-B20)	11.03 ±1.14	8.10–12.94	2.1246 ±0.94	0.9445-2.909
Anti-scale (A1-A5)	2.15 ±1.09	1.25-4.04	1.0246 ±0.028	0.9986-1.07149
Kitchen (K1)	11.38 ±0.18	11.19-11.56	1.0133 ±0.0074	1.0178-1.0174
Drain Open (D1-D2)[a]	12.54±0.071	12.49-12.59	Solid	-

*three separated measurements were carried out for Each sample.

Pappalardo *et al*. reported that there was an association between the values of pH and instability of sodium hypochlorite solution [34]. This solution was more stable at pH 11 and above [18, 35]. In fact, this limit was the same limit set by the Libyan Standard Legislation for the pH of commercial bleach solution containing sodium hypochlorite [30]. In our study, six bleach samples had pH values less than the Libyan Standard Legislation limit as illustrated in figure 1.

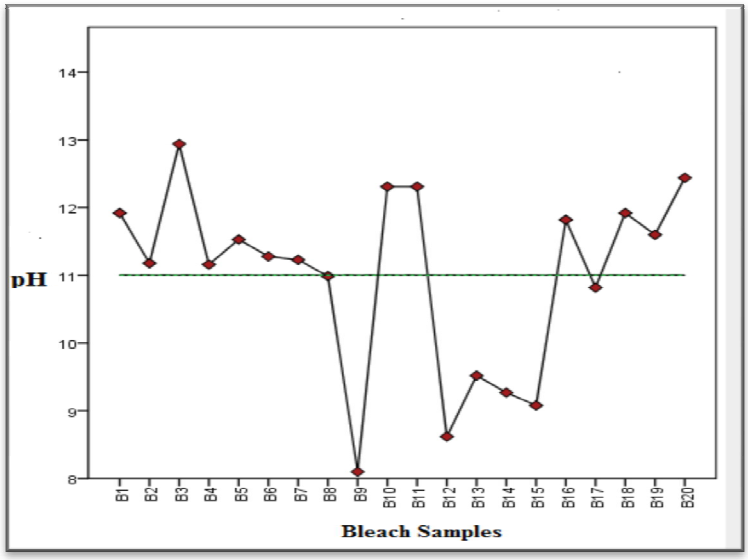


Figure 1. Comparative pH values of bleach cleaning products with the maximum allowed limit set by Libyan standards Legislation (------).

The pH values of anti-scale, kitchen and drain open household cleaners were extremely ranged between strong acidic to strong basic, table 3. The pH values of the anti-scale cleaners were ranged from 1.25± 0.070 to 4.03 ±0.134. In fact, the pH of anti-scale cleaners are usually below 2, because these cleaners contain strong acids such as hydrochloric acid, which can dissolve most mineral scale (mostly silica, calcium carbonate, gypsum, etc) and iron stains [2, 26]. On the other hand, the pH values of the kitchen and drain open cleaners' solutions were corresponding to 11.38 ±0.184 for sample K1, and 12.54 ±0.071 for drain open samples, table 3. These formulations almost contain sodium hydroxide [2, 36].

Determination of the active constituents of Household Cleaning Products: The concentration of sodium hypochlorite and active chlorine contents in the bleach samples were determined byan indirect method (iodometric titration).

The results are recorded in table 4. The sodium hypochlorite content in bleach cleaning samples ranged between 0.0585% and 17.2541%, and the corresponding available chlorine contents ranged from 0.05572% to 16.4324%. In fact, the Libyan Standard Legislation required >5.5% and <7.5% of sodium hypochlorite content, which corresponding to mean value 6.5% (or required >5.25% and <7.125% of available Chlorine content, with the mean value 6.186%), in bleach solutions [30]. In this study, there were 17 bleach products (i.e. 85% of the investigated samples) contained sodium hypochlorite less than 5.5%, which is the minimum limit for the sodium hypochlorite recommended by Libyan standards. Table 3, also shows that three samples (B7, B9, B11) had mean values of the sodium hypochlorite contents far below the limit of Libyan standards. On another hand, sample B3 and sample B16 showed the highest contents of sodium hypochlorite, which corresponding to 12.4538% (available chlorine11.8607%), and 17.2541% (available chlorine 16.4324%), respectively.

Table 4. The Sodium hypochlorite and available chlorine contents of Cleaning Bleach Products

Sample	Sodium hypochlorite (%)[a]	Available chlorine (%)[b]
B1	3.8024 ±0.02	3.6213
B2	4.0842 ±0.08	3.8897
B3	12.4538 ±0.70	11.8607
B4	3.04686 ±0.09	2.9018
B5	2.6604 ±0.16	2.5337
B6	1.5971 ±0.04	1.5211
B7	0.1611 ± 0.05	0.1535
B8	3.3844 ±0.12	3.2233
B9	0.05850 ±0.02	0.05571
B10	3.6783 ±0.31	3.5031
B11	1.9664 ±0.06	1.8728
B12	0.1978 ±0.02	0.1884
B13	1.6119 ±0.05	1.5351
B14	3.2559 ±0.54	3.1008
B15	7.8153 ±0.44	7.4431
B16	17.2541 ±0.44	16.4324
B17	1.97191 ±0.12	1.8780
B18	1.9534 ±0.04	1.8604
B19	2.9145 ±0.03	2.7757
B20	4.4519 ±0.12	4.2400

a Each value is the average of three separated determinations
b these values were calculated using equation (2).

The results in table 4 shows that contents of sodium hypochlorite in B1 and B2 were 3.8024 and 4.0842% and the contents of chlorine were 3.6214% and 3.8897%, respectively. There was no significant difference between sample B1and sample B2 in sodium hypochlorite contents (P=0.195). In fact, these values were below the Libyan Standard Limit [30]. Sample B3, had sodium hypochlorite content corresponding to 12.4538%. This content was highly above the limit of Libyan Standard. A significant difference in the sodium hypochlorite contents was seen between sample B3 and the other two imported bleach samples (B1 and B2). The intergroup comparison for sodium hypochlorite contents was carried out using LSD, and the results showed that a statistic difference was found between sample B3 with both sample B1 (P=0.00) and sample B2 (P=0.00). The sodium hypochlorite concentration in B3 sample was also extremely high than the specified concentration of sodium hypochlorite on the product labelled. A significant different was detected between the actual sodium hypochlorite concentration in this sample and the label specifications of sodium hypochlorite using one sample t-test (P=0.003).

As shown in table 4, sample B15 contained 7.8153% of sodium hypochlorite, which corresponded to 7.4431% of available chlorine content. The concentration of sodium hypochlorite in sample B15 was similar to maximum limit recommended by Libyan Standard (7.5%) [**30**]. However, the One-Sample t-test indicted, that there was a significant difference in the sodium hypochlorite content of B15 and the maximum limit set by Libyan Standard Legislation (7.5%) [**32**], on another hand, sample B20 contained sodium hypochlorite of 4.4519% (available chlorine content 4.2400%). This concentration was similar to minimum limit recommended by Libyan Standards (5.5%).

The percentage values of total acidity were recorded in table 5. The concentrations of hydrochloric acid in five commercial household anti-scale cleaners ranged between 0.5067% and 8.3313%. In fact, Libyan Standard Legislation required >13% and <25% of hydrochloric acid content, which corresponding to mean value 19% in liquid products of anti-scale cleaners [**31**].

APPLICATION

The results obtained from this study are very important for public health and for citizens' awareness of consuming household products. The excessive use of cleaning products affects human health and the environment.

CONCLUSION

In the present study, twenty eight samples of household cleaning have been analyzed to detect their pH and determine the content of the main component using the suitable chemical methods. The results showed that, the pH values of the cleaning samples were ranged from 8.10-12.94 for bleach samples, 1.25 to 4.04 for anti-scale cleaners, and 11.19 to 12.59 for kitchen and drain open cleaners. Sodium hypochlorite concentrations in bleach cleaners were ranged between 0.0585% -17.2541%. The total acidity% of the anti-scale samples ranged from 0.5067% to 8.331%. While the total alkalinity% of kitchen and drain open cleaners ranged from 2.947% to 100.0%, respectively. Furthermore some samples had active constituents much higher than the maximum limit recommended by Libyan Standard Legislations, and another samples their concentration of the active gradients were less than the minimum limits set by Libyan Standard Legislation. Therefore, the consumers are at risk of acute and chronic exposure to the cleaning products. On the base of the results of this study and keeping in view the harmful effects caused by the components of the cleaning household products, it is highly recommended that there should be a regulatory system appointed by government to achieve routine analysis to monitor the quality of cleaning household products purchased at the markets.

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