

# Post-Consumer Polyethylene Terephthalate (PET) Recycling in Bangladesh through Optimization of Hot Washing Parameters

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## Abstract

Most of the Post-Consumer polyethylene terephthalate (pcrPET) is recycled conventionally in Bangladesh and subsequently produces inferior quality recycled PET flakes. Rest of the unused pcrPET is directly goes to the landfills which creates potential environmental hazard for Bangladesh. To get rid of environmental hazard and produce quality recycled PET flakes, pcrPET was mechanically recycled through hot washing. In hot washing different caustic soda and detergent dosage were used for pcrPET flakes effective decontamination. Later, spectrophotometric analysis and contamination test were carried out for recycled PET flakes characterization. Both of the analysis along with quality analysis confirmed together 3.0 percent caustic soda and 0.5 percent detergent dosage in hot washing at 90 °C temperature produce quality recycled PET flakes. Above optimum hot washing parameters effectively removed contaminants from pcrPET waste and finally produced quality recycled PET flakes for successive applications.

**Keywords:** PET recycling; Hot washing; Spectrophotometric analysis; Contamination test; Polyethylene terephthalate.

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## 1. Introduction

Solid waste generation is accompanied with the economic development of a country. Therefore, developed countries have highest solid waste generation rate than the developing countries [1, 2]. However, due to rapid economic growth, now even developing countries are also producing more solid waste than expected [1].

As a result, green revolution movement started to stop potential environmental threat from increasing solid waste as it polluted environment in many different ways [3]. Firstly, indiscriminate disposal scatters solid waste in water network as well in drainage system which consequently disrupts ecological balance and water congestion across the region [1, 4]. Moreover, it also hampers aesthetic appearance of a habitable area since it is not uncommon that solid waste is littered around the dumping area [5]. Without this indiscriminate solid waste disposal also occupies habitable land results scarcity of accommodation for growing population across the world [1, 5]. Furthermore, green house gas generation and contamination in food chain from solid waste compel researchers and concerned authorities to drive research toward the effective solid waste management [4, 6]. Moreover, the Environmental Protection Agency (EPA) has defined plastic waste as one fifth of the total solid waste and this non bio degradable fraction is increasing further [3, 7]. For this large fraction of non bio degradable plastic materials, recycling has become the most attractive and pragmatic research for its management [2, 8]. Across the world from developing to developed countries, lots of investment and research are being carried out for plastic waste recycling. Even communities are also merged with plastic waste recycling program closely in developed countries [1, 9].

With a varying opinion in existing literature, Post-Consumer PET (pcrPET) waste is considered as the second largest portion after poly ethylene waste in plastic waste stream [9]. This huge amount of pcrPET waste generation is due to its wide spread use in textile industry, high strength fiber production, photographic film production, packaging industry mainly in beverage bottles production as an alternative of PVC and glass bottles [3, 9, 10]. Widespread use of PET creates severe ecological problems as a part of solid waste which drives pcrPET recycling across USA, Africa, and Europe to Asia [11, 12]. For example, USA recycled 649 million ton pcrPET in 1997 and Japan increased its pcrPET waste recycling from 2 percent in 1995 to 70 percent in 2007 [3, 8]. Above PET recycling lowers the burden of solid waste as well acts for conservation of both petrochemical products and energy for virgin PET resin production [10]. Moreover, pcrPET recycling is more attractive in developing countries as revenue is associated in every step of recycling from collecting, sorting to final production [1]. For instance, in 2015 Bangladesh used all together 75,000 metric tons (MT) plastic raw materials and recycled around 65 percent of its Post-Consumer plastic materials which was increasing in later years [13].

Broadly PET recycling can be carried out as following way: pre-consumer PET waste recycling, mechanical recycling, chemical recycling and energy recovery through incineration [14, 15]. Among them although first option is most favorable, it does not require any further research as pre-consumer waste does not have any contamination to be removed. As a result it can be recycled within the in plant technology with virgin PET resin [14]. However, recycling by incineration is not also favorable due to strict particulate emission and green house gas generation laws [1, 3]. Therefore, chemical recycling in glycolysis, aminolysis, acidolysis, and hydrolysis route can be carried out for polymer building block monomer production, as an example, pcrPET waste is

recycled in glycolysis route into UV curable acrylate/methacrylate monomer [3, 16]. Furthermore, pcrPET was blended with different polymer and co-polymer for improved thermal and physico-mechanical property as a part of waste PET recycling [10]. However, above PET recycling technology and chemical recycling still requires further technical exploration and private-public investment for their commercial application. On the contrary, mechanical pcrPET recycling involves initial sorting, grinding, decontamination, melt extrusion, and finally pelletizing [14, 17]. It also involves lower capital costs and mostly favorable for developing countries as it requires only effective decontamination process during recycling. For effective decontamination during mechanical recycling, caustic soda and or detergent were used at different dosage in pcrPET washing [15]. In Brazil, only 5M caustic soda is using at elevated temperature for pcrPET decontamination [18]. Moreover, Welle, F. and Park and his colleagues [15, 19] suggested to use both caustic soda and detergent dosage in pcrPET washing for effective decontamination. However, above varying chemical dosage does not effective in Bangladesh for pcrPET recycling due to its different collection method and post treatment after PET materials first hand use [20]. Currently, In Bangladesh, pcrPET is recycled by using a small dosage of detergent which subsequently produces inferior quality recycled PET flakes [21]. As a result, yet no big plastic conglomerates have used recycled materials, only small and medium-sized enterprises (SMEs) used 60 percent of recycled materials in 2010 [13, 22]. Rest of the unused and recycled materials was exported to outside the country due to lack of proper decontamination technology [22]. Therefore, this paper finds out optimum caustic soda and detergent dosage as well as washing temperature for pcrPET flakes washing (termed as hot washing) by considering country's own conditions. Above optimum hot washing parameters enabled pcrPET flakes effective decontamination and subsequently produced quality recycled PET flakes.

## **2. Methodology**

### ***2.1. PcrPET sample collection and preparation***

PcrPET sample was collected from waste bins and landfills which also contained other plastic wastes and metals. All other wastes were separated from pcrPET through manual sorting.

Then pcrPET washing with water at ambient condition removed loosely attached and bulk amount of dirt from it. Initial washing provided relatively clean pcrPET which later cut down into 6–8 mm sized PET flakes.

After that density separation in a water bucket separated polyolefin plastic materials and finally provided pcrPET flakes for hot washing. This pcrPET flakes contained mostly glue and dirt contaminants which were removed in hot washing.

### ***2.2. Hot washing of pcrPET flakes***

Hot washing used chemical dosage at elevated temperature to remove contaminants from pcrPET flakes in a reactor known as hot washer.

Both caustic soda and detergent at different percentages were employed in hot washer. Two hot washers with same specifications are operated in series manner for effective decontamination.

**Table 1:** Specifications of hot washer

Specifications	Value
Effective volume	1 Liter
Stirrer	200 rpm
Sample size	166.67 gm
Residence time for sample	20 minutes

For decontamination, pcrPET flakes hot washing conditions were changed accordingly Table 2 to find out optimum hot washing parameters. Both caustic soda and detergent dosage percentages were counted as percentage of hot washer effective volume (1L).

**Table 2:** pcrPET flakes hot washing recipe

Parameters	Hot washing conditions			
	Caustic dosage (percentage)		Detergent dosage (percentage)	Temperature (°C)
Caustic soda and detergent dosage optimization	0.5 – 6.0		0.25	80
	(0.5 increment)	percent	0.50	
			0.75	
Temperature optimization	Optimum caustic soda and detergent dosage			70 – 90 (increment 10)

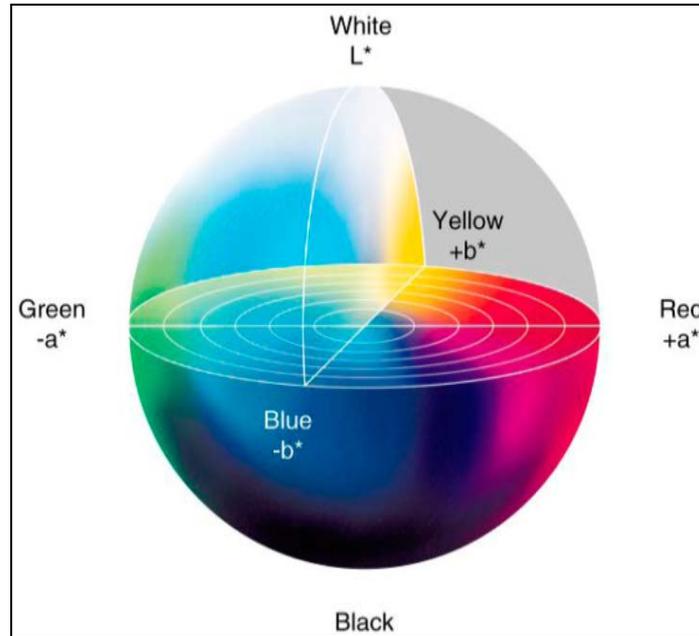
For instance, caustic soda dosage was gradually increased from 0.5 percent to 6.0 percent at a constant detergent dosage. Three different constant detergent dosage values were selected and those were 0.25, 0.5 and 0.75 percent. Each time hot washing used different caustic soda dosage at a constant detergent dosage to find out optimum chemical dosage.

### 2.3. PcrPET flakes washing and drying

After pcrPET flakes hot washing vigorous washing steps such as centrifugal washing, float washing and spray washing were employed one after another for at least 10 minutes for hot washing chemicals removal. After that flakes were dried for 5 minutes at 120 °C for water removal. Flakes drying produced quality recycled PET flakes which were characterized by spectrophotometric analysis and QT500 contamination test.

**2.4. Recycled PET flakes spectrophotometric analysis**

A reflectance spectrophotometer KONIKA MINOLTA CM- 2500d measured recycled PET flakes color parameters in a three dimensional space (Figure 1). As well as their corresponding meanings are listed in Table 3 during recycled PET flakes characterization.



**Figure 1:** L\*a\*b\* color space measurement [adapted from <https://largeinkjet.wordpress.com/tag/lch/>]

**Table 3:** CIELAB color space measurement for recycled PET flakes characterization

Color parameters	Value	Color	Meaning
L*	0	Darkest black	Ineffective removal of dirt contamination from pcrPET flakes in hot washing
	100	Perfect white	Effective dirt contamination removal.
b*	(-)Ve	Blue	Effective glue contamination removal and proper use of hot washing chemicals.
	(+)Ve	Yellow	Deposition of hot washing chemicals on flakes surface and ineffective glue contamination removal during hot washing.
a*	(-)Ve	Green	Effective flakes decontamination
	(+)Ve	Red	Ineffective flakes decontamination

**Table 4:** Contaminants color after QT500 test

Contamination	Color
Hot washing chemicals and additives	
Glue	
PVC	
Blackish flakes	Slightly Black

**2.5. Recycled PET flakes contamination test: QUICK TEST QT500**

Amount of different contaminants in recycled PET flakes were measured according to QUICK TEST QT500. A certain amount of recycled flakes were heated in oven at 220°C for half an hour during QT500 test. As a result, contaminants present in recycled PET flakes showed distinctive color as Table 4 and finally expressed their amounts in ppm (mg kg<sup>-1</sup>) unit.

Both hot washing chemicals and additives (used during PET production) present in recycled flakes turned into yellow colored flakes. However, additives contamination could not remove through hot washing and it remained in all recycle PET flakes sample. Therefore, it was excluded from yellow colored contamination to calculate hot washing chemicals contamination. Exact amount of hot washing chemicals contamination was calculated by comparing yellow colored contamination of recycled PET flakes against pure PET flakes.

## **2.6. Recycled PET flakes quality parameters measurement**

Recycled PET flakes color parameters and amount of different contaminants were measured by spectrophotometric analysis and QT500 test respectively. Besides, intrinsic viscosity was measured according to ASTM D4603 method. Moreover, Differential Scanning Calorimeter DSC 2910 measured recycled PET flakes melting point. Without this AXIS ATS60 moisture meter measured recycled flakes water content and its size was measured manually.

## **3. Results and discussion**

### **3.1. Chemical dosage optimization for pcrPET flakes hot washing**

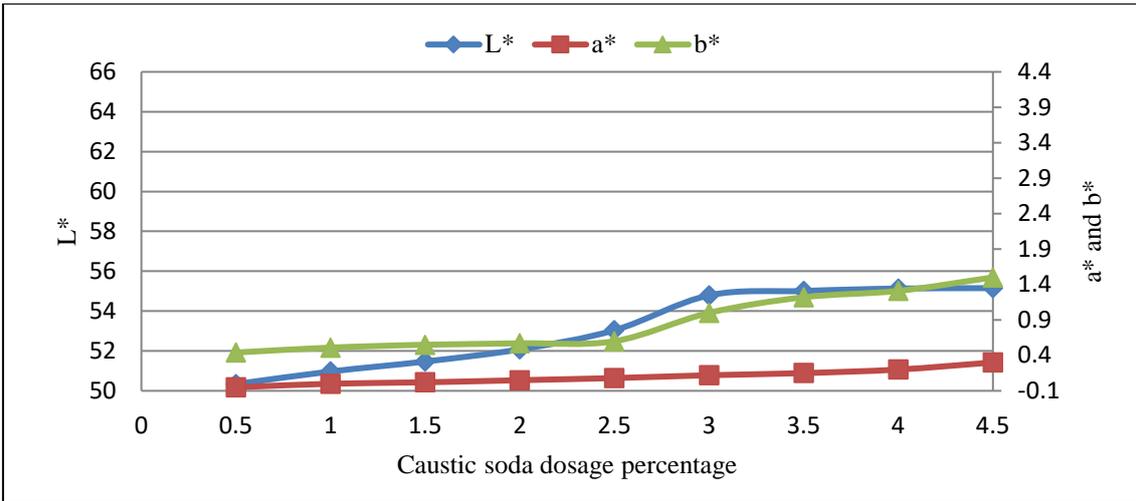
Varying amount of caustic soda and detergent dosage were employed at hot washer to find out optimum chemical dosage for pcrPET flakes hot washing. PcrPET flakes hot washing used 0.5 to 6.0 percent caustic soda at constant 0.25, 0.50, and 0.75 percent detergent dosage. Finally, both spectrophotometric analysis and contamination test of recycled PET flakes determined optimum chemical dosage for pcrPET flakes effective decontamination through hot washing.

#### **3.1. a. Spectrophotometric analysis of recycled PET flakes produced through varying caustic soda and detergent dosage at hot washing**

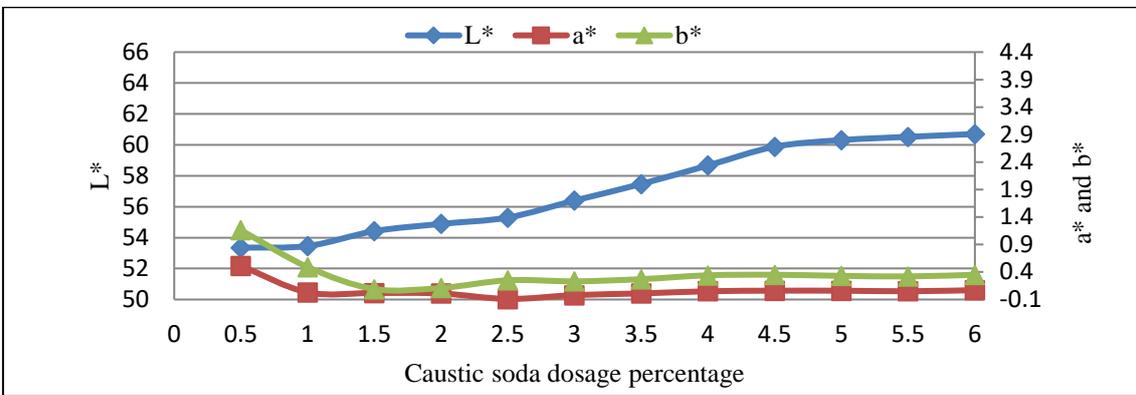
PcrPET flakes hot washing started with relatively lower percentage of constant detergent dosage, 0.25 percent, while caustic soda dosage were changing from 0.5 to 4.5 percent. Spectrophotometric analysis of recycled PET flakes, produced by such chemical dosage, represented in Figure 2 (a). It showed that lower percentage of detergent dosage resulted gradual increase of  $L^*$  value from 50.34 to around 55.0 with caustic soda dosage increase up to 3.0 percent. After that  $L^*$  value did not increase gradually even after subsequent increase of caustic soda dosage up to 4.5 percent rather  $L^*$  value remained constant at around 55.0. This trend was observed due to rapid increase of both  $a^*$  and  $b^*$  value after 3.0 percent caustic soda dosage which slowed down increment of  $L^*$  value at higher caustic soda dosage.

Therefore, detergent dosage was increased to 0.5 percent. This increased usage of detergent dosage influenced  $L^*$  value rapidly from initial point of caustic dosage to final dosage point, i.e., in Figure 2 (b),  $L^*$  value increased rapidly from 53.35 to 60.7 within 0.5 to 6.0 percent caustic dosage. Moreover, other color parameters  $a^*$  and  $b^*$  also reduced initially and then remained stable at 0.05 and 0.35 respectively with increased caustic dosage.

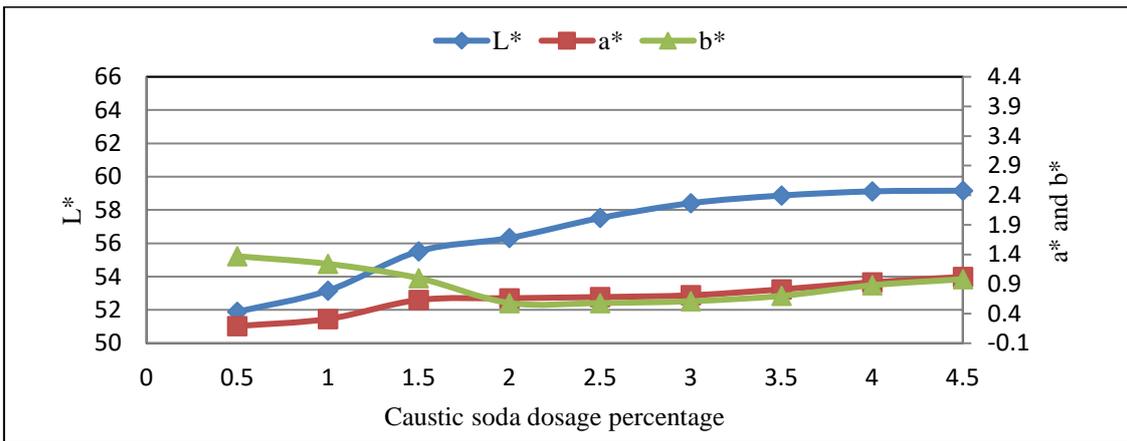
Later detergent dosage increased further to 0.75 percent to investigate whether this increment could increase effectiveness of hot washing or not for pcrPET flakes decontamination. Spectrophotometric analysis of recycled flakes (Figure 2 (c)) showed an initial increase in  $L^*$  value but later it remained stable at around 59.0 with increased percentage of caustic soda dosage. As well as other color parameters  $a^*$  and  $b^*$  also increased gradually with caustic soda dosing increment.



(a)



(b)



(c)

**Figure 2:** Spectrophotometric analysis at varying caustic and (a) 0.25 percent (b) 0.5 percent (c) 0.75 percent detergent dosage.

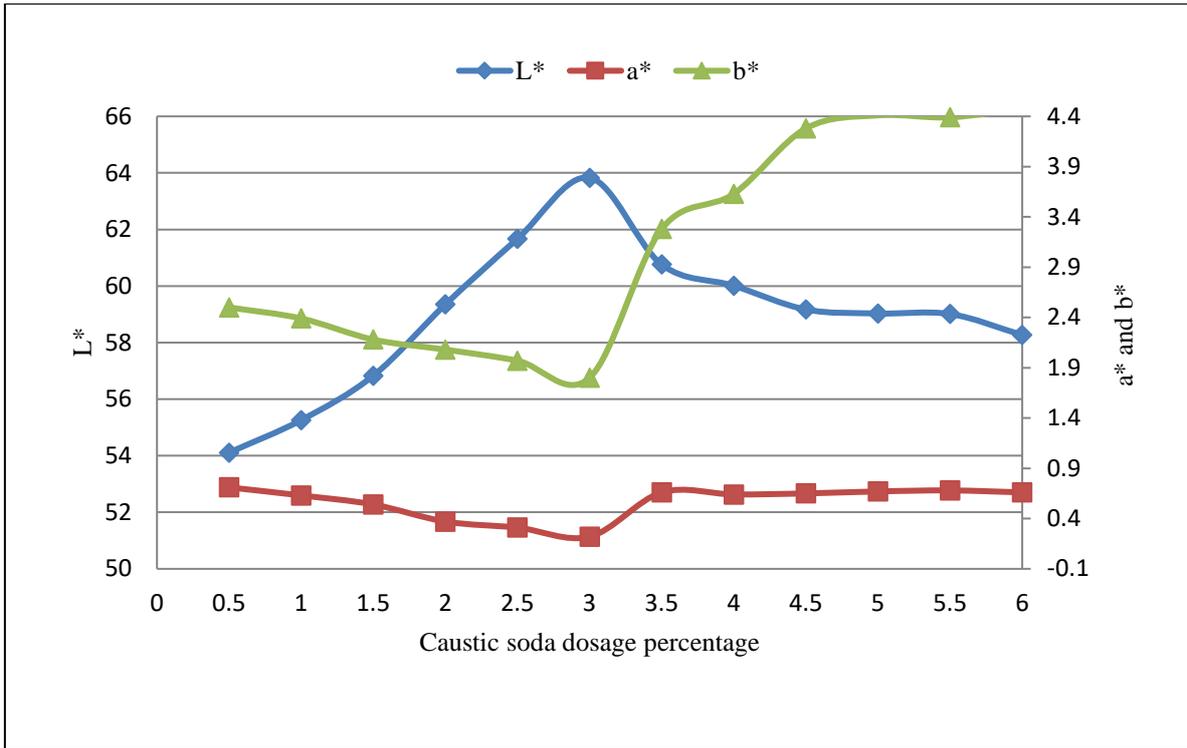
Spectrophotometric analysis concluded that lower percentage of detergent dosage (0.25 percent) could not decontaminate pcrPET flakes effectively even at higher percentage of caustic soda dosage. This led to increased usage of detergent dosage at 0.50 percent. Spectrophotometric analysis at this higher detergent and caustic soda dosage ensured effective decontamination of pcrPET flakes through hot washing. Further detergent dosage increment at 0.75 percent could not improve hot washing effectiveness for decontamination within 0.5 to 4.5 percent caustic dosage range. This was mainly because of higher percentage of chemicals (mainly detergent) dosage at hot washing was not removed at subsequent vigorous washing steps. As a result caustic soda and detergent remained on flakes surface and resulted higher  $a^*$  and  $b^*$  value and lower  $L^*$  value. Therefore, 0.5 percent detergent along with 0.5 – 6.0 percent caustic soda was fixed as optimum chemical dosage for pcrPET flakes hot washing.

### ***3.1. b. Contamination test of recycled PET flakes produced through varying caustic soda and 0.5 percent detergent dosage at hot washing***

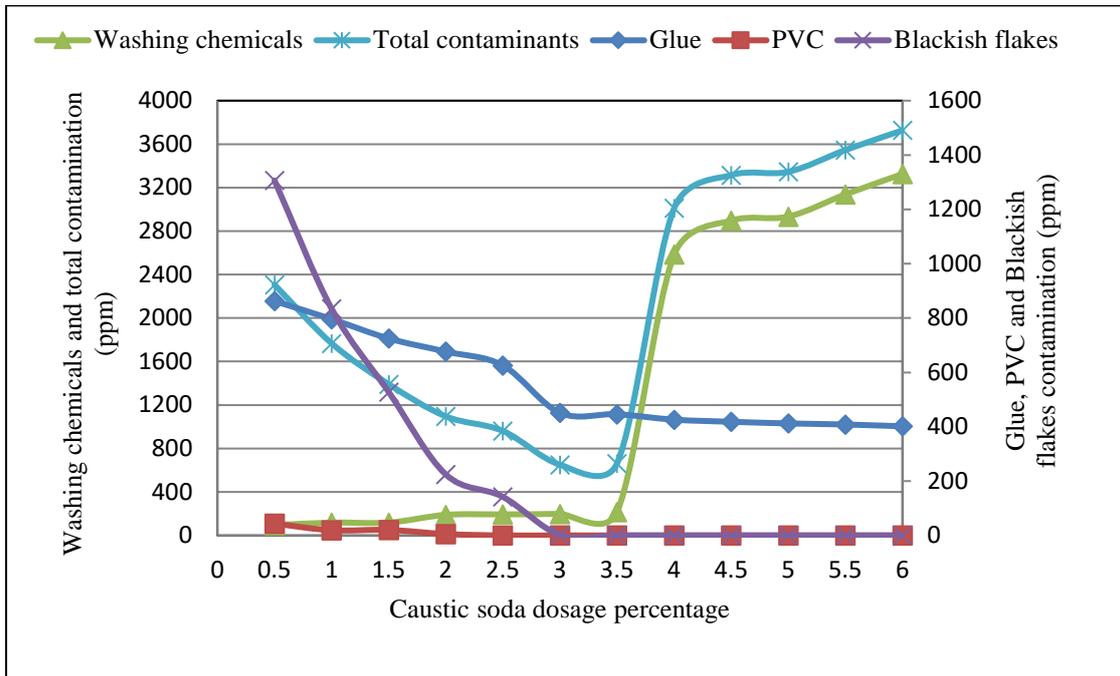
Spectrophotometric analysis at Figure 2 (b) ensured higher percentage of caustic soda dosage effectively removed contaminants from pcrPET flakes at hot washing. But it involved higher operating costs from purchasing higher amount of caustic soda. As well as utilization of higher caustic soda effects total contamination of pcrPET flakes. Both of those led the contamination test of recycled PET flakes to determine optimum caustic soda dosage from 0.5 – 6.0 percent at 0.5 percent detergent dosage for hot washing. Contamination test measured amount of different contaminants in recycled PET flakes, simultaneously, flakes color parameters were monitored by spectrophotometric analysis.

Spectrophotometric analysis of recycled flakes (Figure 3 (a)) after contamination test showed a rapid increase in  $L^*$  value with caustic soda dosage increase and a highest peak in  $L^*$  value (63.83) observed at 3.0 percent caustic soda dosage. Besides a gradual decrease observed in both  $a^*$  and  $b^*$  values up to this caustic dosage. Later,  $L^*$  values decreased rapidly and both  $a^*$  and  $b^*$  values increased with the increase of caustic dosage up to 6.0 percent. Those later trends in color parameters indicated presence of unused hot washing chemicals, caustic soda and detergent, in PET flakes surface. It also confirmed from gradual increment of  $b^*$  value up to 4.48 at 6.0 percent caustic dosage.

Moreover, deposition of unused hot washing chemicals on PET flakes surface beyond 3.0 percent caustic soda dosage also confirmed from contamination test by its distinctive color shown in Table 4. Contamination test result of recycled PET flakes represented in Figure 3 (b). Among different contaminations, amount of washing chemicals contamination in PET flakes increased dramatically from 198.58 ppm at 3.0 percent caustic dosage to 3325.42 ppm at 6.0 percent caustic dosage. This washing chemicals contamination significantly increased total amount of contaminants after 3.0 percent caustic dosage as blackish flakes and PVC contaminants were removed fully before this caustic dosage. Actually, total contamination was summation of all other contaminants: washing chemicals, glue, PVC, and blackish flakes contamination. Therefore, 3.0 percent caustic soda and 0.5 percent detergent dosage fixed as optimum chemical dosage for hot washing as this dosage contained lowest amount of total contaminants 651.05 ppm among which 69.23 percent was glue contaminant.



(a)



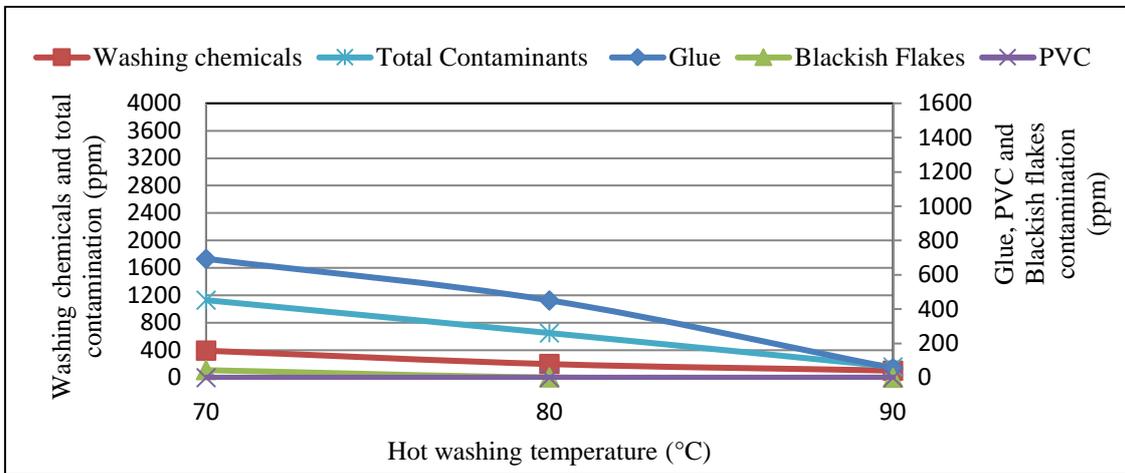
(b)

**Figure 3:** Recycled PET flakes (a) spectrophotometric analysis and (b) contamination test within 0.5 – 6.0 percent caustic soda and 0.5 percent detergent dosage.

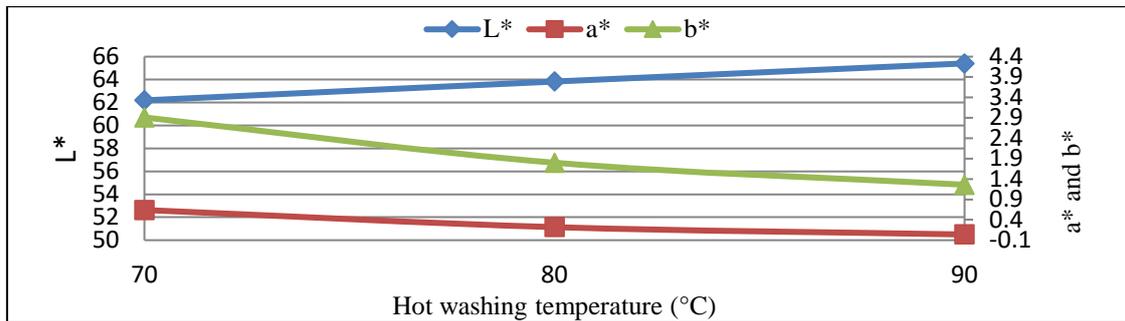
**3.2. Hot washing temperature optimization for glue and other contaminants removal**

Optimum hot washing chemical dosage, 3.0 percent caustic soda and 0.5 percent detergent dosage, although contained lowest amount of total contamination, it still had 69.23 percent glue contamination. This glue contamination mainly as well as hot washing chemicals and blackish flakes contamination was minimized through alteration of hot washing temperature. As mentioned earlier all the previous hot washing was carried out at 80 °C temperature which resulted, for example, only 30.77 percent glue contamination removal at 3.0 percent caustic dosing. Therefore, at optimum chemical dosage, hot washing temperature was changed from 70 °C to 90 °C but kept it below the boiling point of water.

Figure 4(a) represented hot washing temperature increment at 90 °C reduced glue contamination at 56.06 ppm from 451.04 ppm of 80 °C. It also reduced amount of both unused hot washing chemicals and blackish flakes contamination at 103.56 ppm and 0 ppm respectively and subsequently decreased total contamination from 651.05 ppm to 159.62 ppm. As a result, total contamination in recycled PET flakes reached at lowest amount 159.62 ppm with optimum 3.0 percent caustic soda and 0.5 percent detergent dosage in hot washing.



(a)



(b)

**Figure 4:** Recycled PET flakes (a) contamination test (b) spectrophotometric analysis at varying hot washing temperature

Moreover, increased hot washing temperature (90 °C) also affected color parameters. Previous L\* value 63.83

at 80°C increased up to 65.41 as well as both a\* and b\* value also decreased as shown in spectrophotometric analysis in Figure 4(b). Therefore, increased temperature 90 °C at optimum chemical dosage was the optimum hot washing temperature. Optimum hot washing temperature provided following two fold advantages: amount of glue as well as total contamination reduction and color parameters improvement.

**3.3. Recycled PET flakes quality analysis**

Quality parameters of recycled PET flakes, produced through optimum hot washing parameters: 3.0 percent caustic soda and 0.5 percent detergent dosage at 90°C temperature, tabulated in Table 5. For a comparative study, quality parameters tabulated against recycled PET flakes specifications [17]. Table 5 showed that PVC, Metal and Polyolefin contamination level was less than specified values due to effective manual sorting and density separation. However, some other major contaminants do not exist in specification although they have been used extensively nowadays for recycled PET flakes quality control [5, 19]. For example, glue contamination is the major reason for recycled PET flakes yellow coloration but it does not have any specification [5, 24]. However, optimum hot washing parameters reduced glue contamination at 56.06 ppm. Moreover, as this paper decontaminated pcrPET through chemical dosage in hot washing, some of unused hot washing chemicals remain in final recycled flakes. And optimum chemical dosage had minimum amount of hot washing chemicals contamination, 103.56 ppm, at 90 °C temperature. Similarly, optimum hot washing parameters resulted lowest amount of total contamination as well. Moreover, previously recycled PET flakes color content was monitored by yellowing index, but nowadays CIE L\*a\*b\* color space measurement is widely used in many PET recycling regulatory bodies and research activities [18, 23, 25]. Table 5 showed that optimum hot washing parameters produced recycled PET flakes with better color properties than specifications. Moreover, comparative study in Table 5 showed other quality parameters, for example, intrinsic viscosity, melting point, moisture content etc. also met the quality specifications. As a result, finally it can be concluded that optimum hot washing parameters produced quality recycled PET flakes.

**Table 5:** Recycled PET flakes quality parameters

Quality parameters	Specification	Recycled PET flakes
Polyolefin contamination	<10 ppm [17]	0 ppm
PVC contamination [a]	<50 ppm [17]	0 ppm
Metal content	<3 ppm [17]	0 ppm
Glue contamination [b]	Not specified	56.06 ppm
Washing chemicals contamination [c]	Not specified	103.56 ppm
Blackish flakes contamination [d]	Not specified	0 ppm
Total contamination [e] [e]=[a]+[b]+[c]+[d]	Not specified	159.62 ppm
Color parameters	L*	60 – 62 [23]
	a*	Not specified
	b*	3.0 – 3.3 [23]
Intrinsic Viscosity	0.7 dl gm <sup>-1</sup> [17]	0.71 dl gm <sup>-1</sup>
Melting point	>240 °C [17]	247.23 °C
Water content	<0.02 wt.% [17]	0.01 wt.%
Flake size	0.4 mm < D < 8 mm [17]	6 - 8 mm
Dye content	<10 ppm [17]	Not measured

#### 4. Conclusion

Lower percentage of detergent dosage, 0.25 percent, along with 0.5 – 6.0 percentage of caustic dosage cannot wash pcrPET flakes properly and subsequently contaminants remain in recycled PET flakes. On the contrary, at the same conditions higher amount of detergent dosage, 0.75 percent, deposits hot washing chemicals on PET flakes surface results undesired color parameters (lower L\* value and higher a\* and b\* value). In between them 0.5 percent detergent dosage effectively washes pcrPET flakes for decontamination. Later, contamination test confirms deposition of hot washing chemicals on PET flakes beyond 3.0 percent caustic soda dosage at 0.25 percent detergent which also results higher amount of total contaminants in recycled PET flakes. As a result, 3.0 percent caustic soda along with 0.5 percent detergent is taken as optimum chemical dosage for effective decontamination in hot washing. Finally, hot washing temperature 90 °C at above optimum chemical dosages effectively removes mainly glue as well as blackish flakes and hot washing chemicals contamination and subsequently results lowest amount of total contamination in recycled PET flakes. Therefore, optimum chemical dosage at 90 °C is the optimum parameters for pcrPET flakes hot washing. Before mentioned optimum hot washing parameters produce quality recycled PET flakes which was confirmed in quality analysis against specifications. Production of quality recycled PET flakes firstly set up a systematic procedure for pcrPET waste recycling in Bangladesh. Secondly, as now quality flakes have been produced, it does not necessary to export inferior quality recycled flakes anymore. Instead quality recycled flakes can be used to meet demand of big plastic conglomerates of the country for their packaging applications. As a result, amount of virgin PET polymer resin import will be reduced as well as SMEs can now produce quality products from quality recycled PET flakes and can turn their venture into a profitable one. However, pcrPET sample was collected from all the major cities of Bangladesh to homogenize the PET waste generated across the country. Most of the small cities of the country were excluded during pcrPET sample collection. Therefore, to fully apply the recycling procedure adopted in this paper to recycle the pcrPET waste, sample should be collected from all the cities of Bangladesh.

#### 5. Recommendations

Although quality recycled PET flakes produced in this paper as per quality specifications, further tests, for example, end group analysis, jar test etc. should be carried out before their application in food packaging and pharmaceutical industry.

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