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Treatment Effects of Pavement Milling Materials of Semi-rigid Base with Acid Solution Method

Luxin Fu[1], ZhirongJia[2], Ruirui Jiang[3] and Lizhi Wang[4]
[1]*School of Transportation and Vehicle Engineering*
[2]*School of Civil and Architecture Engineering*
[3]*School of Transportation and Vehicle Engineering*
[4]*School of Transportation and Vehicle Engineering*
Shandong University of Technology, Zibo, CHINA
Corresponding Author: ZhirongJia.

ABSTRACT: *Highway overhaul and reconstruction engineering produce a large amount of semi-rigid base milling material every year. In order to improve the physical and mechanical performances of milling coarse aggregates, and increase added value of waste pavement materials. The SEM and EDS tests were used to analyze the microstructure and chemical composition of milling coarse aggregates of the cement stabilized aggregate (CSA) and the lime-fly ash stabilized aggregate (LFSA). The recycled coarse aggregates were soaked with 0.1mol hydrochloric acid and acetic acid solution respectively. Mechanisms of acid treatment were analyzed, and the crush value and water absorption of coarse aggregate were tested. The results showed that the crushing values of CSA and LFSA milling materials with hydrochloric acid were reduced by 7.6% and 3.6%, and the water absorption rates were reduced by 0.9% and 3.3% respectively; the crushing values were reduced by 4.3% and 27% with acetic acid, and water absorption rates were reduced by 0.5% and 1.7% respectively. The adhesion mortars of milling aggregates can be effectively removed by soaked with two kinds of acidic solutions, and aggregates performances can be improved. But the hydrochloric acid treatment effects are better than that of acetic acid.*

KEYWORDS: *milling material; cement stabilized aggregate; lime-fly ash stabilized aggregate; acid treatment; physical and mechanical performances*

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I. INTRODUCTION

China's road engineering construction is in a period of rapid development presently. However, rigorous environmental protection measures have greatly reduced the quarrying of building materials, and the huge demand for building materials in construction projects has caused road building materials to become increasingly scarce. In order to solve the contradiction between environmental problems and aggregate demand, material recycling has attracted more and more attention.

Since the 1920s, some developed countries, such as Germany, Japan, Netherlands and America, have developed rapidly in resource recycling, and have achieved a series of achievements. Recycled aggregates are divided into various types according to waste materials, such as waste concrete (Anon, 1983; Chen et al., 2003), waste asphalt (Taha et al., 2002), waste bricks (Khalaf, 2006), used tires(Moo-Young, 2003) etc.. In the early stage, scholars studied the application of RCA in concrete structures, and explored the preparation of recycled concrete by adopting different dosage of RCA and different water cement ratio (Hansen, 1986; Ryu, 2002； Xiao, et, al., 2006; Yamasaki, et al., 1998, etc). With the development of society, the requirement of recycled concrete in engineering construction is increasing. In order to prepare high quality recycled concrete, scholars begin to study the factors that affect the performance of recycled concrete. Katz (2004) proposed that RCA covered with loose particles is the main reason to prevent the good combination between new cement matrix and recycled aggregate, and then affects the performance of recycled concrete. Cho (2011) pointed out that the cement slurry adhered to the surface of RCA and the low-density, high-porosity pollutants are the main reasons for the poor quality of RCA engineering and are not recommended for hot mix asphalt mixture. From these studies, it can be found that the porous and micro-crack weak layer formed by cement slurry attached to the surface of recycled aggregate is the main reason that affects the strength and performance of recycled aggregate concrete, and is also one of the most significant differences between recycled aggregate and natural aggregate. Although the use of recycled aggregate can reduce the consumption of limited resources and consequently protect the environment and save costs, there are still some shortcomings in the use of recycled aggregate, such as the weak interfacial ability between recycled aggregate and cement slurry, and the cement slurry attached to the surface of recycled aggregate leads to the lower quality of recycled aggregate (Prokopski et al. 2000; Ollivier et al. 1995; Tam et al. 2005). The limitation of recycled aggregate application is its poor quality.

In order to improve the utilization value of RCA, many scholars have studied how to improve the quality of RCA. According to the different methods of treating RCA surface slurry, the research can be divided into two categories. One category is to modify the RCA surface. Katkhuda (2017) improved the mechanical properties of RCA by adding 0.1%, 0.3%, 0.5%, 1% and 1.5% chopped basalt fiber (BF). Katz (2004) treated recycled aggregate by impregnating silica fume solution. Wang (2017) improved recycled aggregates quality based on bio-deposition of bacteria-induced CaCO3 precipitation. Jingwei(2017) used high concentration of CO2 gas to strengthen RCA. Haili(2004) soaked RCA with 3% water glass solution. HouYueqin et al. (2013) used silicone resin to activate RCA. The other category is to remove cement slurry on the RCA surface. Qiuyi (2011) used particle shaping method to shape RCA particles. Tsujino (2008) removed the slurry on the RAC surface by heating and grinding the recycled aggregate. The above method for removing the slurry has problems such as complicated process flow, difficulty in realization, and high energy consumption, and the promotion is limited. Chemical treatment method for removing the slurry is a new attempt. Ismail (2014) treated RAC by using different acid molarities at low concentration as agent for surface treatment. Purushothaman (2014) and Tam (2007) pre-soaked RCA with a certain concentration of hydrochloric acid, sulfuric acid, and phosphoric acid solutions obtained RCA with low water absorption and other desired properties. However, these studies on the reinforcement of recycled aggregates are mostly based on RAC, but rarely based on recycled aggregates of semi-rigid base materials.

In highway construction, the semi-rigid base materials are mainly cement stabilized macadam and lime-fly ash stabilized macadam. It is one of the effective ways for road construction projects to take a sustainable development path. However, the traditional treatment methods for semi-rigid base layer milling materials include discarding directly, filling roadbed or in-situ cold regeneration, etc.. These treatment methods result in low added value of semi-rigid base layer milling materials. In order to improve its added value, it is necessary to explore a suitable strengthening treatment method for semi-rigid base milling materials from improving its quality.

In this paper, CSA and LFSA milling materials are treated by the acid cleaning method, and the physical and mechanical properties of the two milling materials in these two acidic environments are studied and analyzed, and for the domestic road construction specification requirements, suggestions on the application of the two types of grass-roots milling materials used in road applications are put forward.

II. MATERIALS AND METHODS

2.1 Materials

The milling material used in the experiment was CSA milling material and LFSA milling material obtained by Wirtgen W2000 milling machine, as shown in Fig. 1

(**a**) (**b**)

Figure 1: Surface morphology of milling material of CSA(a) and LFSA(b)

The coarse aggregate with a particle size of more than 4.75 mm was sieved by a sieving machine. The Properties of CSA and LFSA milling material are shown in Tab. 2. Both hydrochloric acid and acetic acid are chemical products of fine chemical plants in Laiyang Economic and Technological Development Zone. The specific technical indicators are shown in Tab.1.

Table 1: Technical indicators of hydrochloric acid and acetic acid

Properties	Hydrochloric acid	Acetic acid
content(%)	35-38	99.5
crystallization point(℃)	-	≥15.1
evaporation residue(%)	≤0.0005	≤0.002
mixed with water test	eligibility	eligibility
chloride(%)	≤0.0001	≤0.0001
sulfate(%)	≤0.0002	≤0.0002
titanium(%)	-	≤0.0001
copper(%)	≤0.00001	≤0.00005
lead(%)	-	≤0.00005
iron(%)	≤0.00005	-
arsenic(%)	≤0.00005	-
tin(%)	≤0.0002	-
heavy metal(%)	≤0.00002	-
acetic anhydride(%)	-	≤0.2
reductive dichromate substance(%)	-	≤0.008

Table 2: Properties of two base layer milling materials

Properties	CAS milling material	LFSA milling material
apparent density(g/cm^3)	2.70	2.69
needle like content(%)	8	9
water absorption(%)	3.5	8.4
mud content(%)	0.54	0.77
crushing value(%)	31.2	34.2

2.2 Test design

2.2.1 SEM microscopic scan and EDS analysis

In order to analyze the influence of microstructure and components on the properties of recycled aggregates, SEM microscopic electron microscopy and EDS material element types and contents analysis were performed on the two types of substrate milling materials.

2.2.2 Hydrochloric acid and acetic acid soaking treatment

The milled material was treated with 0.1 mol hydrochloric acid and acetic acid solution. The acid solution treatment process included immersing the milling material in an acidic environment at 20 degrees Celsius for 24 hours, and then removing the acidic solvent on the surface of the milling material by using distilled water, and soaking the washed milling material with clean water for 24 hours. The soaked milled material was dried in a 105-115 degrees Celsius environment. The treated milling material was obtained.
The specific process of acid solution treatment is shown in Fig. 2:

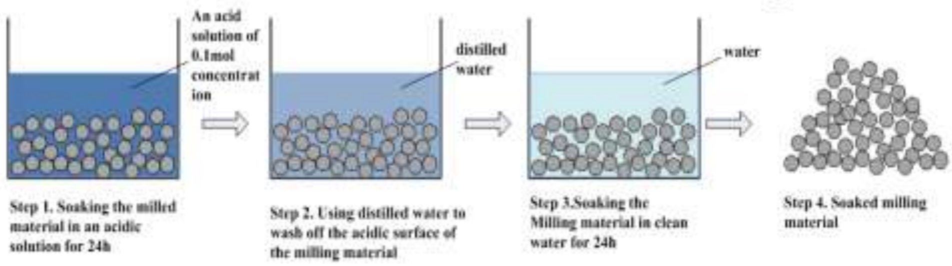


Figure 2: Acid solution treatment process of milling material

2.2.3 Crush value test

The treated milling material was sieved with standard sieves of 13.2mm and 9.5mm. Took 3 sets of 9.5mm to 13.2mm samples, and the mass of each set was 3kg.The sample was divided into 3 times (1kg each time) and the test mold was uniformly loaded. In each case, the surface of the specimen was leveled and the hemispherical end of the metal rod was evenly tamped 25 times from the aggregate surface. Finally, the surface was carefully leveled with a metal bar as a straight blade.

Weighed the sample mass (M_0) in the cylinder. Put the test mold with the sample on the universal testing machine, and put the pressure head on the aggregate surface of the test mold at the same time, paid attention to level the indenter, did not wedge the side wall of the test mold.

Started the machine, and applied the load evenly. The total load was applied to 400 KN in about 10 minutes, and stabilized the pressure for 5 seconds, then unloaded. Removed the test mold from the machine and took the sample out.

All crushed samples were screened with a standard sieve of 2.36 mm, which could be screened for several times until there were no obvious residues in 1 min. weighed all the fines (M_1) that pass through the 2.36 mm sieve, accurate to 1g.
Calculation formula of crush value was Eq.1.

$$Q_1 = \frac{M_1}{M_0} \times 100\% \tag{1}$$

where, M_0-sample mass before test(g); M_1-fine mass passing 2.36 mm sieve after test(g).

2.2.4 Water absorption test

The treated milling material was sieved with standard sieves of 13.2mm and 9.5mm. Took2 sets of 9.5mm to 13.2mm samples, and the mass of each setwas1kg.Put 9.5mm~13.2mm of milling material into clean water and soaked it for 24h, then took out and put it into enamel plate. Wiped the material with a wrung wet towel to absorb the free water leaked from the milling material.

No particles should be lost in this step. Then the surface of the aggregate particles should be gently wiped with a wrung wet towel until there are no shiny water traces on the surface. Keeping the surface dry state, weighed the aggregate apparent mass ($m_{f)}$ immediately.

Placed the milled material in a shallow pan and dried it in an oven at 105°C±5°C until constant weight. Took out the pan, placed it in a container with a lid and cooled it to room temperature, then weighed the aggregate dry mass (m_a).
The same set of milled material was tested twice in parallel, and took the average as the test result. The water absorption was accurate to 0.1%.
Calculation formula of water absorption was Eq.2.

$$\omega_x = \frac{m_f - m_a}{m_a} \times 100 \tag{2}$$

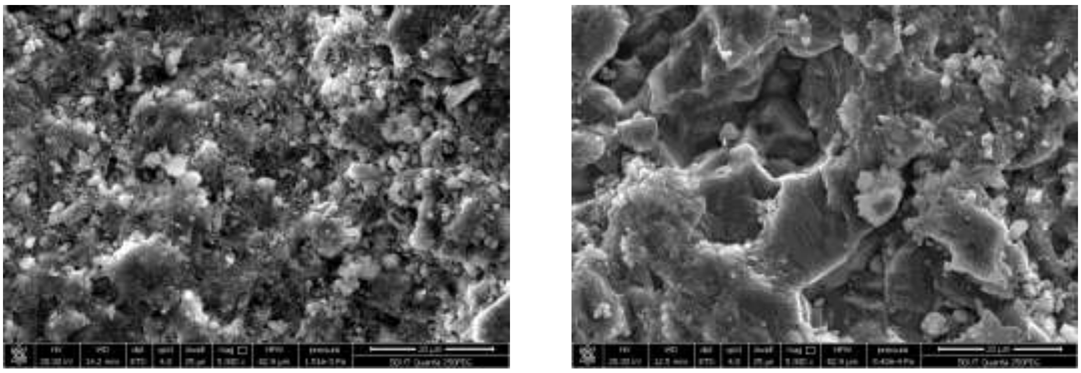
where, ω_x - coarse aggregate water absorption(%); m_a - coarse aggregate drying mass(g) ;
m_f - coarse aggregate surface dry mass (g).

2.3 Test results and analysis

2.3.1 Microscopic morphology and composition analysis of milling materials

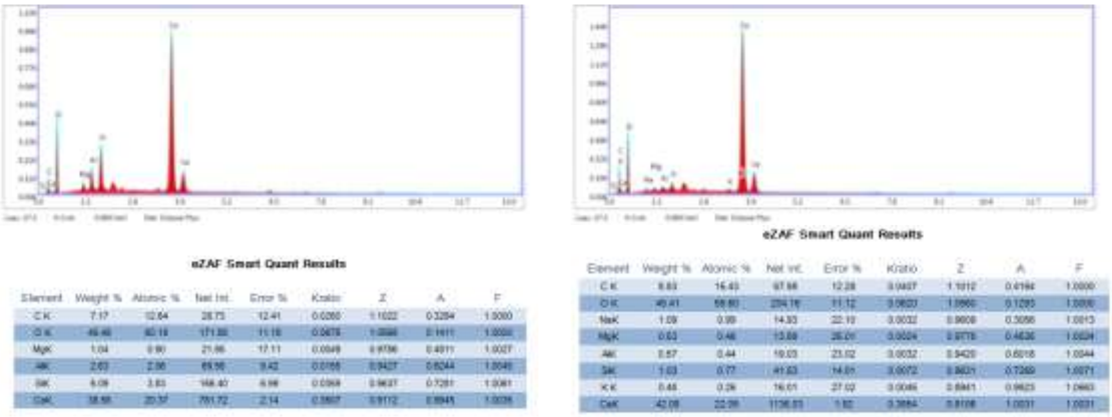
As shown in Fig. 3, a large number of tiny particles are attached to the surface of CSA milling materials, and there are a large number of pores on the surface of LFSA milling materials observed under the 5000x scanning electron microscope. These tiny particles would influence the strength of new CSA and water requirement of new CSA mixture. Existed pores of milling material, especially LFSA, influence the aggregate water absorption. The more pores, the higher the water absorption.

EDSanalysis is performed on the surface of the untreated milling material. From Fig. 4, the main components of the cement mortar and the lime-fly ash mortar on the surface of the milling material are Ca, O, C. The small amounts of elements include Si, Al, Mg, and a small amount of elements K, Na only in the lime-fly ash mortar.



(a) (b)

Figure 3: SEM images(5000x) of CSA(a) and LFSA(b) milling materials



(**a**) (**b**)

Figure 4: EDS analysis of CSA(a) and LFSA(b) milling materials

The mortar main components of CSA and LFSA covered by the surface of the milling material include calcium oxide (CaO), silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), Magnesium oxide (MgO), Unresolved calcium carbonate ($CaCO_3$) , sodium oxide (Na_2O), and Potassium oxide(K_2O).According to the proportion of the elements in the EDS analysis, the content of each main component in the mortar can be obtained, as shown in Tab. 3.

Table 3: The main components of the mortar on the surface of the milling material

Indicators	Content(%)	
	CSA milling material	**LFSA milling material**
CaO	66.8	74.4
SiO_2	11.6	2.3
Al_2O_3	3.0	0.6
$CaCO_3$	16.3	19.8
MgO	2.4	1.2
K_2O	-	0.5
Na_2O	-	1.2

2.3.2 Microscopic morphology and composition analysis of milling materials

The surface morphology of the milling material after acid treatment is shown in Fig. 5.

After the acid leaching treatment, the performance indicators of the two types of milling material are shown in Tab. 4.

Tab. 2 shows that the water absorption of CSA milling material is 3.5%, the water absorption of LFSA milling material is 8.4%, and that of the natural aggregate is less than 1.1%.The higher water absorption of milling material is due to that the natural aggregate adhered old mortar. The presence of old mortar makes the milling material have a higher porosity and therefore have a higher water absorption.

(**a**) (**b**)

Figure 5: CSA(a) and LFSA(b) milling materials treated with acid

Table 4: Performance of the milling material before and after acid treatment

Properties	Milling material	Before the acid leaching treatment	After the acid leaching treatment	
			Hydrochloric acid （HCL）	Acetic acid （CH_3COOH ）
crushing value (%)	CSA	31.2	23.6	26.9
	LFSA	34.2	30.6	31.5
water absorption(%)	CSA	3.5	2.6	3.0
	LFSA	8.4	5.1	6.7

The main components of the old mortar include calcium oxide (CaO), silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and magnesium oxide (MgO).In an acidic environment (H+), water-soluble compounds are generated, so the acidic solution removes most of the attached mortar.

From Tab. 4, it can be seen that the crushing value and water absorption are significantly reduced after pretreatment of the milling material. The crushing values of the hydrochloric acid-treated CSA milling material and the LFSA milling material are reduced by 7.6% and 3.6% respectively, and the water absorption rates are reduced by 0.9% and 3.3% respectively. After acetic acid treatment, the crushing values are reduced by 4.3% and 2.7% respectively, and the water absorption rates are reduced by 0.5% and 1.7% respectively.

This shows that the acidic solution treatment can effectively remove most of the old mortar from the milling material and help to improve the weak layer of the milling material.

2.3.3 Mechanism Analysis

From Tab. 3, it can be found that the main components of the old mortar covered the milling material include calcium oxide (CaO), silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and magnesium oxide (MgO). The reactions in the acidic environment of hydrochloric acid and acetic acid are shown as chemical Eq.3~Eq.10. $Ca(CH_3COO)_2$, $Mg(CH_3COO)_2$ and $Al(CH_3COO)_3$ are unstable and soluble in water, so the acidic solution can remove most of the attached old mortar.

The reactions under hydrochloric acid (HCl) are shown as chemical Eq.3~Eq.6.

$2HCl+CaO \rightarrow CaCl_2+H_2O$, (3)

$2HCl+CaCO_3 \rightarrow CaCl_2+H_2O+CO_2\uparrow$, (4)

$2HCl+MgO \rightarrow MgCl_2+H_2O$, (5)

$6HCl+Al_2O_3 \rightarrow 2AlCl_3+3H_2O$, (6)

The reactions under acetic acid (CH3COOH) are shown as chemical Eq.7~Eq.10.

$2CH_3COOH +CaO \rightarrow Ca(CH_3COO)_2+H_2O$, (7)

$2CH_3COOH +CaCO_3 \rightarrow Ca(CH_3COO)_2+H_2O+CO_2\uparrow$, (8)

$2CH_3COOH +MgO \rightarrow Mg(CH_3COO)_2+H_2O$, (9)

$6CH_3COOH +Al_2O_3 \rightarrow 2Al(CH_3COO)_3+3H_2O$, (10)

By treating the two kinds of base layer milling materials with an acidic solution, a large amount of old mortars attached to the milling material surface are washed away, and the quality of the milling materials are improved.

After acidic solution treatment, the chloride and sulfate content of the milling material increased, but still within the standard range of 0.06% and 2% respectively. Regarding the pH value, the milling materials treated with hydrochloric acid and acetic acid are still in the alkaline range (pH 8.5 or higher), indicating that the milling materials have lesser resistance to the acid treatment.

2.4 Application analysis of milling coarse aggregate

`The general requirements for the mechanical and physical properties of reclaimed aggregates are shown in Tab. 5 according to the highway engineering standard(JTG/T F20-2015).

From Tab. 4 and Tab. 5, it can be seen that the crushing value is reduced to below 30%, and the water absorption is also reduced to 3% after CSA milling material are treated with hydrochloric acid and acetic acid. Reclaimed coarse aggregates meet the basic requirements of road base and sub-base.

The crushing value and water absorption of LFSA milling material treated by hydrochloric acid and acetic acid solution are reduced, but the requirements for the coarse aggregate are not met in the standard of the road base and sub-base, so it cannot be used in the construction of the base and sub-base of the highway engineering.

Table 5: The performance requirements of coarse aggregate

Road grade	Pavement structure	Properties of coarse aggregates	Requirements	
			Stabilized aggregate	Graded aggregate
secondary and lower than secondary roads	base	crushing value	≤35%	≤30%
		water absorption	≤3%	≤3%
	sub-base	crushing value	≤40%	≤35%
		water absorption	≤3%	≤3%

III. DISCUSSION AND CONCLUSION

Acid solution cleaning method can effectively remove the adhered mortar of milling coarse aggregates.

The crushing value and water absorption of the two kinds of milling materials decrease after soaked with hydrochloric acid and acetic acid.

The mechanical and physical properties of reclaimed coarse aggregate of CSA milling material can meet the basic requirements of road base and sub-base after treated by hydrochloric acid and acetic acid solution.

The crushing value and water absorption of reclaimed coarse aggregate of LFSA milling material are reduced treated with hydrochloric acid and acetic acid. But it does not meet the standard requirements of road base and sub-base, and cannot be used in the construction of road base and sub-base of the road engineering.

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